DEVELOPMENT OF A PRESSURE MOULDING SYSTEM TO FABRICATE FRP PRODUCTS

by
B. RAVINDRANATHA REDDY

DEPARTMENT OF MECHANICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY KANPUR

JANUARY 1997

DEVELOPMENT OF A PRESSURE MOULDING SYSTEM TO FABRICATE FRP PRODUCTS

A Thesis Submitted
in Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

by

B . RAVINDRANATHA REDDY

to the

Department of Mechanical Engineering
INDIAN INSTITUTE OF TECHNOLOGY KANPUR

JANUARY, 1997

CERTIFICATE

It is certified that the work contained in the thesis entitled **" DEVELOPMENT OF A PRESSURE MOULDING SYSTEM TO FABRICATE FRP PRODUCTS "**, by **B.Ravindarnatha Reddy** , has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

P Kumar

(Prof. Prashant Kumar)
Department of Mechanical Engineering
Indian Institute of Technology, Kanpur

January, 1997

ACKNOWLEDGEMENTS

I express my deep sense of gratitude and indebtedness to Dr. Prashant Kumar for his invaluable guidance and constructive suggestions during the present work.

I wish to thank Mr. Anurag Goel, Mr. R.C.Tiwari and Mr.B.D.Pandey for thier help in preparing the experimental set up and specimens. Special thanks to Mr. Pankaj Singh Chandel for his help and co-operation throughout my work.

I am very grateful to all friends, especially A.Ramakrishna, for helping me in one way or other and making my stay in Kanpur a memorable one.

B.Ravindranatha Reddy

Dedicated to

My Parents

and

Sister

CONTENTS

CHAPTER 2

Pressure Moulding System

CHAPTER 3

Product Characterization and Discussion

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

A **Pressure Moulding System** has been developed to fabricate some products of Fibre Reinforced Polymer material. Three different products and required tooling have been developed. E-Glass fabric in woven form and Epoxy resin are used in the present work. Pressures ranging from 0.4 MPa to 0.7 MPa and maximum cure temperature of 120°C were used. The products fabricated using this system are: Circular cross-sectioned tube, Convex-concave surfaced tube, and Circular tube with square groove. The circular tube and convex-concave surfaced tube were tested for dimensional stability and were found to be dimensionally stable. For characterization of circular tube, fibre volume fraction, flexural strength and modulus of elasticity were determined. The average flexure strength and average modulus of elasticity obtained were 133.21 MPa and 9.2 Gpa, respectively at average fibre volume fraction of 41.77 %. Tensile test was carried out to compare these results. The average tensile strength and modulus of elasticity were 159.8 MPa and 16.44 GPa, respectively at average volume fraction of 44.15 %.

CHAPTER 1

INTRODUCTION

1.1 FIBRE REINFORCED POLYMERS

Polymer composites reinforced with fibres possess low weight along with high specific strength, stiffness and high-temperature resistance. Their other advantages include ease of processing and structural forms that are otherwise difficult to manufacture. Therefore, they are increasingly used for structural parts in aircraft and space applications, in the automobile industry, and for sporting goods.

1.2 REINFORCEMENTS

The commonly used fibres are glass, carbon and aramid fibres. They are supplied in variety of forms suitable for different applications. Continuous rovings, woven rovings, cloths and random chopped fibre mats are some of the commercially available forms.

Glass fibres are the most widely used reinforcement in the composite industry as they are cheap and the relative ease in their production. E-glass and S-glass are two prominent glass varieties. E-glass exhibits excellent tensile strength ($\sigma_u = 3.5$ GPa) and good electrical properties. S-glass, with a tensile strength 30% greater than E-glass is used for ultra high strength applications. However, low modulus and poor abrasion

resistance limit their usage.

Carbon fibres are the predominant high strength, high modulus reinforcement used in the fabrication of high performance composites. Carbon fibres exists in two forms- diamond or graphite. Carbon fibres of ultra high modulus are produced with higher percentage of diamond structure or fibres of moderate modulus with high percentage of graphite structure. Commercially available carbon fibres have modulus ranging from 27.6 Gpa to 400 Gpa, and tensile strength 0.9 Gpa to 3.1 GPa [Agarwal 1990]. But carbon fibres are the most expensive of the common reinforcements.

Polyaramid fibres offer an unique combination of properties in many applications. The well known aramid fibre in commercial production is Kevlar. The specific strength of Kevlar is very high as its density is much lower than glass or carbon fibre. It has high impact resistance and ballistic energy absorption. But it has low compressive strength. So, by combining with other fibres in the form of hybrid fibres, we can take advantage of their unique properties. This combination of excellent specific strength and modulus performance combined with toughness and impact resistance makes aramid fibre composites an attractive choice in aerospace and satellite applications.

1.3 MATRIX MATERIALS

The most common matrix materials for fibrous composites are the organic 'super polymer' materials, commonly called plastics. Good chemical resistance, ease in processing, low specific gravity and tailorability of properties are some of the advantages of polymers. There are essentially two types of plastics: thermoset and thermoplastics. Thermoplastics readily flow under stress at elevated temperature, and they can be processed into the required shape of a component. These polymers may be repeatedly heated, fabricated and cooled and consequently scrap may be recycled, though their properties slightly degrade. Well known thermoplastics include acrylic, nylon (polyamide), polyethylene, and polystyrene.

Thermosetting polymers are resins which readily cross-link during curing and harden irreversibly. Epoxides, polyesters, phenolics are some of the commonly used thermosetting polymers. Epoxy resins,though expensive, are very commonly used for high load bearing applications.

1.4 FABRICATION OF FIBROUS COMPOSITES.

The fabrication of polymer composites into finished products involves the combination of the matrix and fibre such that the matrix impregnates and wets the fibre. An efficient fabrication process should ensure uniform resin distribution and good wetting of fibres, with very few void contents [Lubin 1982]. These things mainly depend on the curing temperature and pressure. Processes, where curing is carried out at room temperature and with out pressure, are relatively cheaper as they do not require rigid mould, heating oven or pressuring tools. In non-pressure processes, there is no way to remove excess resin and entrapped air, resulting in components with high void content and low volume fraction. These processes are least preferred as primary structural members because they produce components with poor mechanical properties. A moderately good fabrication technique involves room-temperature curing under pressure. For the best results, the curing process is accomplished by exposing the material to elevated temperatures and pressure for a predetermined length of time. The elevated temperatures applied during the cure, make the matrix less viscous and improves wetting of fibres. The applied pressure provides the force needed to squeeze excess resin out of the material, to consolidate individual plies, and to compress vapour bubbles.

The choice of a fabrication process is also influenced by the chemical nature of the matrix- thermoset or thermoplastic, and the temperature required to cure the matrix. Fabrication of thermoplastic matrix composites involves material formation in first stage and moulding a shape in a second operation. The principal method used for the production of fibre reinforced thermoplastics is injection moulding. In case of thermosetting matrix composites, material formation is accomplished during final moulding due to their curing characteristics. They become hard when cured, and further heating doesn't soften them.

Fabrication processes for thermosetting resin matrix composites can be broadly classified as Wet forming processes and Dry forming processes using premixes. In wet forming processes compounding (combining fibres and matrix) and curing is carried in one step. These include hand lay-up, filament winding,pultrusion, and bag moulding. In dry forming processes, compounding (premixing) is done in first step and then lay-up is carried out in second step. These two processes are uncoupled. Examples of these processes are hand lay up, matched-die moulding and bagging methods.

1.5 WET FORMING PROCESSES

1.5.1 Hand Lay-up

Hand lay-up [Hollaway 1994] is the widely used laminating process because of its inherent flexibility and low capital outlay in moulds and tooling. Only a single mould is needed for lay-up. In its basic form resin is mixed in a bucket and applied by brush or mohair roller to the mould. Normally a neat resin layer called the 'gel coat' is applied first and allowed to gel before proceeding with reinforced layers. Alternate layers of resin and reinforcement are then applied to the mould and a ribbed metal roller used to consolidate the laminate and fully impregnate the reinforcement, while at the same time forcing the air out. Large or thick components are usually made in several stages, allowing the resin to gel after each stage before proceeding with the next. Both continuous and discontinuous fibre reinforced components can be laid up. This process gives only one finished surface and is labour intensive, resulting in very low production volume.

In spray-up, chopped fibre from air-driven chopper unit and resin are sprayed on to the mould simultaneously by a special spray gun. This method is best suited to large or medium sized components with no fine detail. The production rate is high. The fibre

orientation, being random in this process, does not make good use of the fibre properties. Thickness control is very poor because, it is an operator dependent process.

1.5.2 Filament Winding

High speed precise winding of continuous fibres or rovings in predescribed patterns over a mandrel is the principle of the filament winding technique. In wet winding, the fibres are impregnated in a resin bath before winding over the rotating mandrel. The winding angles and the placement of the fibres are controlled through specially designed machines, traversing at speeds synchronized with the mandrel rotation. The main advantages of this technique are high production rate due to process automation and good fibre placement control. But this method fails to produce complex shapes and the external surface produced is poor. This method is used for the manufacture of surfaces of revolution such as pipes, tubes, cylinders and spheres. Pressure vessels, tanks, motors, gas bottles are some of the components produced by this method.

1.5.3 Pultrution

This technique is similar to extrusion process except for a few things. The pultrution process consists of drawing continuous fibre rovings or woven mats through a resin bath and then into preforming fixtures where excess resin and entrapped air are removed. Then the composite passes into a heated die where it is cured continuously. The length of heated die and the speed at which the composite passes through the die depend on the curing time of the composite.

Pultrution enables high fibre contents. The tension applied to the fibres ensures good longitudinal alignment and therefore very high and consistent longitudinal mechanical properties can be attained. Pultrution process is used to mass-manufacture composite materials into continuous, constant-cross-section profiles such as rods, tubes, and various structural shapes.

1.5.4 Injection Moulding

The most important of the traditional thermoplastic process used to make composite material is injection moulding. This process is used to fabricate smaller sized parts in mass production.

The raw material for moulding is prepared by compounding the resin and fibres prior to the injection moulding. This compounding can be made in two ways - extruder compounding and strand coating. In the extruder-compounding process,the chopped fibres and resin are fed directly into an extruder for mixing. The later method consists of passing the rovings of fibres through a specially designed extruder die-head so that they are coated and partially impregnated by the molten polymer. The palletized compound is melted (usually in an extruder that forms a part of the injection-moulding machine) and the molten plastic is then injected into a closed mould. The part is mechanically ejected after solidification.

Complex shaped components with very close tolerances can be fabricated at high production rates. But, Tooling costs are very high for this processes. Only short, random

fibre reinforced composites can be produced by this processes.

1.6 DRY FORMING PROCESSES

In dry forming processes, as mentioned earlier, compounding is uncoupled from lay-up. The first step is to make preimpregnated reinforcement (prepreg). Prepreg is made by impregnating fibre rovings or woven fabric in resin and then partially curing it. Under the action of heat and pressure the prepreg forms to the shape of the mould and then sets and cures. Prepreg handling is easy and also components of consistent quality can be produced. In hand lay-up and filament winding techniques, prepregs can be used instead of wet fibres. Some of the dry forming processes are explained below.

1.6.1 Matched-Die Or Compression Moulding

In this process, the equipment is a press (usually hydraulically driven) that is fitted with both male and female dies. The prepegs are laid up on the male die and the mould halves are then pressed together and heated to the cure the part. The pressures developed can range up to several megapascals, which is useful for obtaining good part uniformity and compression of the voids that may develop. Products of complex shapes, good mechanical properties and close part tolerances are possible. High production volumes can only compensate the costly equipment and tooling.

1.6.2 Vacuum-Bag Moulding

Vacuum-bags allow for the production of large, high quality composite parts that cannot be fabricated by mechanical or hydraulic press. In this method, prepregs are laid up on the prescribed pattern and the bagging material(cellophane, polyvinyl alcohol or nylon) is installed over it, by pressing or sealing it to the tape sealant around the mould periphery. The vacuum exit or port is installed and connected, and the air is drawn from beneath the bag. The application of vacuum provides the dual advantage of pressing the layers together and simultaneously withdrawing the excess resin and entrapped air. The pressures usually vary between 69 - 90 kPa.

This process is suitable for fabricating large components which does not need high structural properties. Bagging of complex shapes is difficult and labour intensive.

1.6.3 Pressure-Bag Moulding

Pressure bag moulding is a similar process to vacuum bag and allows the use of higher pressures than atmospheric (up to 0.35 MPa), producing better consolidation and higher fibre contents. Better curing can also be achieved by using heated air in the bag.

The disadvantage of the pressure bag moulding is that the tooling is expensive, and it can only be used for the specific part for which it is designed.

1.6.4 Autoclave Moulding

Autoclave processing is the method of curing or thermal forming a composite under gas pressure greater than atmospheric. The composite part is laid up and enclosed in the vacuum bag. Full or partial vacuum is drawn within the bag, and is cured in an autoclave by simultaneous application of heat and pressure much greater than atmospheric (up to 2 MPa). Application of vacuum at initial stages remove the entrapped air from inside of the bag, while the external pressure provides the mechanical force needed to squeeze excess resin out of the material, to consolidate individual plies, and to compress vapour bubbles.

Dense and large parts with very low void contents can be fabricated using this process. The major difficulty with autoclave moulding is the high capital cost, and very long cure cycles.

1.7 NEED FOR AN ALTERNATIVE TECHNIQUE TO AUTOCLAVE PROCESSING

Hand lay-up processes and Press forming techniques can be used to produce less expensive structural components, but are limited to parts of small to medium size only. Furthermore, their application to parts of complex geometry is difficult and may lead to loss of details. On the other hand, the only presently available technology to fabricate large parts of complex geometry and high degree of integration seems to be the application of autoclave techniques. Latter processes are economically prohibitive so

that their wide spread application to series production is not foreseeable in the near future. There is an urgent need for low cost techniques suitable to produce good quality components.

In this work, a system has been developed based on the Pressure Moulding System , which can produce complex and highly integrated components with acceptable accuracy, using cheap manufacturing lay-ups and tooling. The technique combines low-cost hand lay-up process with ''cheap'' autoclave variants (high pressure, high temperature).

1.8 OUTLINE OF THE PRESENT WORK

The basic principle, design of tooling and other details of Pressure Moulding System have been dealt with in Chapter 2. Chapter 3 presents description of various products produced by this method. The characterization of the products fabricated using this technique has been covered in Chapter 4. The work has been concluded in Chapter 5 with suggestions for future work.

CHAPTER 2

PRESSURE MOULDING SYSTEM

2.1 PRELUDE

The large growth of polymer composites is predicted to continue expanding from aerospace to more commercial areas. The factors hindering their widespread application in engineering and commercial applications are high cost of raw materials, complex and expensive tooling, and high rejection due to inconsistency in quality. All these factors make composite material expensive than conventional metals. In aerospace and military applications cost is of secondary importance, whereas in other engineering applications tooling cost and material cost are given vital importance. The challenge facing the industry has changed and new emphasis is on developing fabrication techniques which reduce cost, maintain or improve quality.

2.2 COMPONENTS PRODUCED BY PRESSURE MOULDING SYSTEM.

A wide variety of products can be fabricated by using pressure moulding system. Separate tooling is needed for each product. In the present work three types of products are developed. They are:

1 Circular Tube

2 Tube with Convex-concave surface and

3 Circular tube with square groove, resembling a key-way.

The circular tube, as shown in Fig. 2.1, is moulded at the beginning to develop the whole system. Then convex-concave surfaced tube (Fig. 2.2) is developed, which showed that negative curvatures can also be produced. Circular tube with square groove (Fig.2.3) throws light on the limitations of mouldability of complex geometries using this method.

2.3 PRINCIPLE OF PRESSURE MOULDING SYSTEM

In pressure moulding system developed in this work, a rubber tube is used to apply pressure on the laminates stacked in an open mould. The fibres are laid up and impregnated in the mould, and pressure is applied by inflating the rubber tube. The inflated tube exerts the required mechanical force to consolidate the plies, and makes excess resin to bleed out. Curing is carried out at desired temperature for a predetermined length of time.

The principle of this system is schematically shown in Fig. 2.4. Basically it consists of a single mould and a rubber tube. The geometry of the mould corresponds to that of the product being fabricated. Usually a cylindrical rubber tube can be used for various cross-sections . For better results the geometry of the tube should be custom designed to suit the shape of the products. Fibres are impregnated with resin and are laid up on the mould surface, and the rubber tube put in place. Metallic plugs are inserted at both ends of the tube and are clamped tightly. Then the rubber tube is inflated, which applies pressure on the laminates. Curing can be done at elevated temperatures by placing the mould in hot-oven, oil-bath, or water-bath.

As curing is carried out at high temperatures and pressures, dense parts with low void contents can be produced. Fibres in all forms can be laid up. This method is suitable to mould axi-symmetric components like tubes of various cross-section, flywheels, without incurring heavily in tooling cost. Components with negative curvature can be moulded easily by this method.

2.4 LABORATORYSCALE SET-UP

Laboratory scale set-up for the pressure moulding system is shown in the Fig 2.5. The photographs of the experimental set-up are shown in Fig 2.6 and Fig 2.7. The main components of the lab scale set-up are the mould, rubber tube, top plate, bottom support plate, and on-line pressure gauge. Details of each component are given in subsequent sections.

2.5 THE MOULD.

2.5.1 For Circular Tube

A hollow cylinder made up of mild steel whose inner surface is finely machined and finished, is used as the mould. Figure 2.8 shows the cross-section of the mould. A draft of 2.5° is provided to facilitate easy removal of the product from the mould after curing. If the length of product is more and the product design does not allow draft, an eject mechanism can be provided or a split-mould designed. However, this problem can be avoided by using a good releasing agent.

At both ends, an undercut of 3 X 17.5 is provided, for the following reasons. The rubber tube when inflated, has minimum radius of curvature of about 2.5 mm. Without an undercut at both ends, the top and bottom portion of about 2.5 mm of the product is not subjected to desired pressure. Provision of this undercut moves the radius of curvature of the tube 3 mm away from the mould surface on either sides. Four holes of M 8 are made on 72 mm pitch circle diameter on either faces of the mould.

2.5.2 For a tube with convex-concave surface

To produce a tube with convex-concave surface, an insert (Fig. 2.9) which has a contour corresponding to the geometry of the product is fitted to the original mould, by using a screw. Figure 2.10 shows the sectional view of the mould with the insert.

2.5.3 For a tube with square groove

An insert with square cross-section (Fig. 2.11) is fixed to the cylindrical mould by using a screw. The whole system now functions as mould for the production of a tube with square groove. The mould along with the insert is shown in Fig. 2.12.

2.6 THE BOTTOM-PLATE

It is a circular plate of mild steel as shown in Fig. 2.13. The function of this plate is to clamp one end of the rubber tube and also as bottom rest to the mould. It has four holes of M 9 on 72 mm pitch circle diameter. The bottom-plate is fixed to the mould by using four screws through these holes. An under cut is provided for proper resting of the mould and to place a gas-ket.

The clamping mechanism is shown in Fig 2.14. A tapered plug is slipped into the tube upto its full-length. This plug is of mild steel with 15 mm long rear portion for easy handling. For easy slipping of the plug into the tube, negative taper of 5 is provided at rear end. A tapered hole which matches to the geometry of the plug is made in the bottom-plate. The plug is seated in this hole, and tightly clamped to the plate by using a screw. When the tube is inflated, the pressure on top-surface of the plug applies an additional clamping force on the rubber tube. Thus, this unit works as a self-locking clamp.

2.7 THE TOP-PLATE

The geometry of the top-plate (Fig 2.15) is similar to that of the bottom-plate except for a few additional features. A mild steel tube is welded to the tapered plug (Fig. 2.16), through which gas is pumped into the rubber tube. The other end of the tube is threaded to fit into a three-way adopter. A hollow screw (Fig 2.17) is used to clamp the tapered plug to its seat such that, the long steel tube passes through the inner hole (Fig.2.18). The inner through-hole in the screw is made slightly bigger than the diameter of the steel tube , for easy movement of the screw. Here also the clamping mechanism

is self-locking. Photographs of the components and the assembled top-plate are shown in Fig. 2.19 and Fig.2.20 respectively.

2.8 ON-LINE PRESSURE GAUGE

To mount the pressure gauge in the system, a three way adaptor is specially designed as shown in Fig 2.21. This adaptor is fitted to the steel tube as mentioned in the above section. A one-way valve is silver brazed to side hole of the adaptor while,pressure gauge of 1 MPa range is mounted on the top.

2.9 THE RUBBER TUBE

In the present work, an ordinary cycle tube (1 1/2 inch) is used for moulding purpose. It can withstand temperatures as high as 150°C at elevated pressure of 0.8 MPa. At room temperature, the rubber tube is stiff and can not take intricate shapes. It is well known that, when rubber is heated to glass transition temperature, there will be large change in modulus and it becomes relatively soft. To obtain rough estimate of glass transition temperature of the rubber material, an experiment has been carried out and is presented in Appendix-A. The glass transition temperature of the rubber used in this work was found to be 70°C.

This rubber tube can be used to produce circular tubes and tubes with moderately complex contours. For complex shaped product, tube with contour corresponding to the geometry of the product is recommended to get good results.

2.10 MOULDING

2.10.1 PREPARATION FOR MOULDING

The mould is polished with a fine emery paper before moulding and whole set-up is cleaned with acetone to remove any resin left over. Two layers of polyvinyl alcohol are

coated on the mould surface and then a layer of wax is applied over it. This acts as mould release agent and helps in easy removal of the component after curing. Similar coatings are also applied to the inside surface of the top-plate, bottom-plate, and the rubber tube.

2.10.2 MOULDING

Before moulding, pieces of woven glass fabric are cut to the desired length and width. To allow stretching of woven fabric during outward radial motion during compression, the circumferential length of fibres is made up of two pieces. These fibres are impregnated with epoxy resin. The desired thickness is obtained by piling up required number of layers (usually 16 layers to get 3 mm thickness [Uday 1996]). The rubber tube is placed inside the mould and, bottom-plate plug and top-plate plug are slipped into the tube one at each end. These plugs are clamped to their corresponding plates as explained earlier. Both the plates are fixed to the mould with bolts. The rubber tube is inflated to desired pressures, depending upon fibre volume fraction requirements. The excess epoxy settle in the undercuts made at both ends of the mould. Curing is then carried out at desired temperatures.

2.10.3 CURING

The usual processing procedure for thermoset curing is to increase the temperature while maintaining pressure. As with most fluids, the viscosity of the matrix will fall with the temperature rise and then allowing good consolidation. Eventually chemical cross-linking will occur to cause the incipient gelation, and the composite will change from a liquid into a solid and will no longer permit plastic deformation. The temperature at which this phase or structural change occurs is called the dynamic gel temperature. Depending on the resin, this temperature occurs when the matrix is 40-60 % cross-linked [Loos 1983]. The gel temperature is dependent upon the time/thermal history of the material. The time for gelation decreases as the curing temperature increases. Therefore, gelation can be achieved with either long times at moderate temperatures or moderate times at

elevated temperature.

The cure cycle should be such that the following requirements are satisfied:

a) the magnitude of the cure pressure is sufficiently so that all of the excess resin is squeezed out from every ply of the composite before the resin gels at any point inside the composite;
b) the resin is cured uniformly throughout the composite at the end of cure;
c) the cured composite has a low void content; and
d) the composite is cured in the shortest time.

In the present work, room temperature curing is done initially for a few products. High pressure in the range of 0.5 MPa - 0.7 MPa is applied for good consolidation. The type of epoxy resin used is LY 556 and HY 951 of Hindustan Ciba-Geigy, Mumbai. The curing reaction is exothermic, so the temperature of the system increases, causing a small decrease in the viscosity for a short period and then the characteristic increase in viscosity with cross-linking. The typical cure cycle at 20°C is:

gelation starts	60 - 75 minutes
80 % curing	8 hours
100 % curing	24 hours.

The vast majority of epoxy composite parts are cured at elevated temperatures with simultaneous application of pressure. In this work, curing was performed with the cure cycles shown in Fig 2.22. The cure temperature was increased at a constant heating rate from room temperature until the maximum cure temperature was reached. Products were cured at the maximum temperatures of 90°C and 120°C maximum temperatures, at a heating rate of 3°C/min. The whole set-up was placed in a hot-water jacket for 90°C curing and in an oil-bath for 120°C. The temperature was controlled by a temperature controller. The cure time (when a degree of cure of 90% is reached at all points of the composite) depends greatly on the applied cure temperature and on the heating rate. Loos and Springer [1983] have shown that a 16-ply composite cured using

a heat rate of 3°C/min and a maximum cure temperature of 120°C takes a cure time of 180 minutes. The same composite when cured at a maximum temperature of 90°C with the same heating rate, takes a cure time of 300 minutes. Low heating rates are recommended to have uniform temperature distribution resulting in uniform curing at all points in the composite part.

After curing the rubber tube is deflated and the mould is removed. The product is taken out and slight finishing, like trimming is done to maintain the width of products.

2.11 CLOSURE

A system for Pressure Moulding System is developed which overcomes some of the limitations of the existing techniques for the fabrication of composites. Three different components with varying degree of complexity of geometry are fabricated. Curing is carried out at elevated temperatures and pressures.

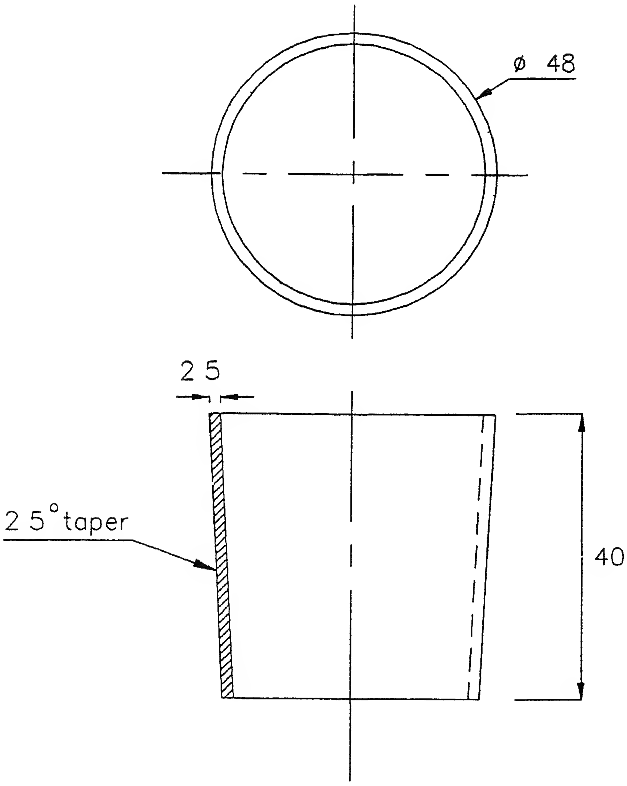


Figure 2 1 Photograph and drawing of Circular tube

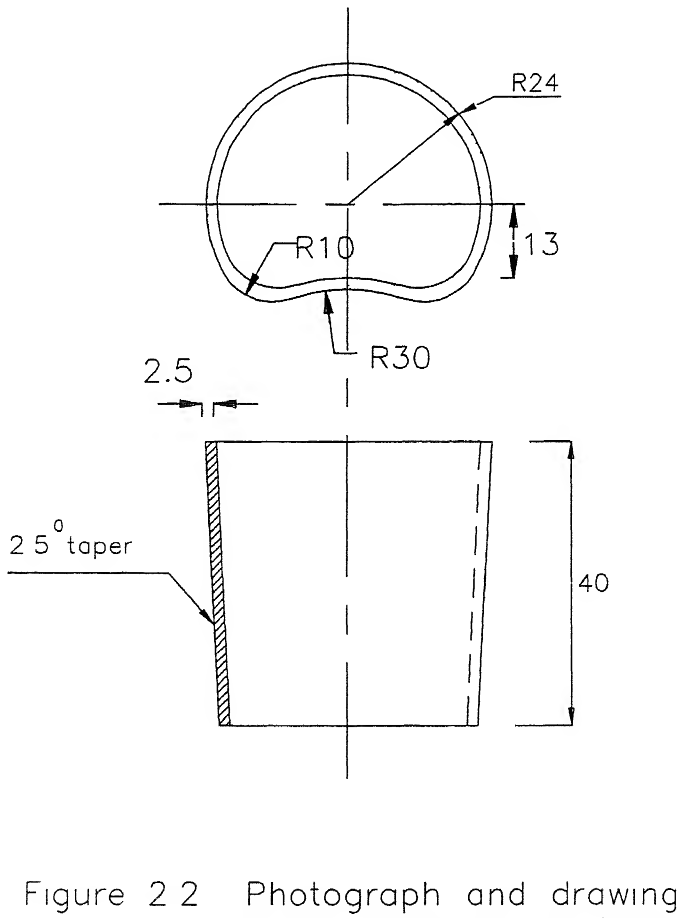


Figure 2 2 Photograph and drawing of Convex–Concave surfaced tube.

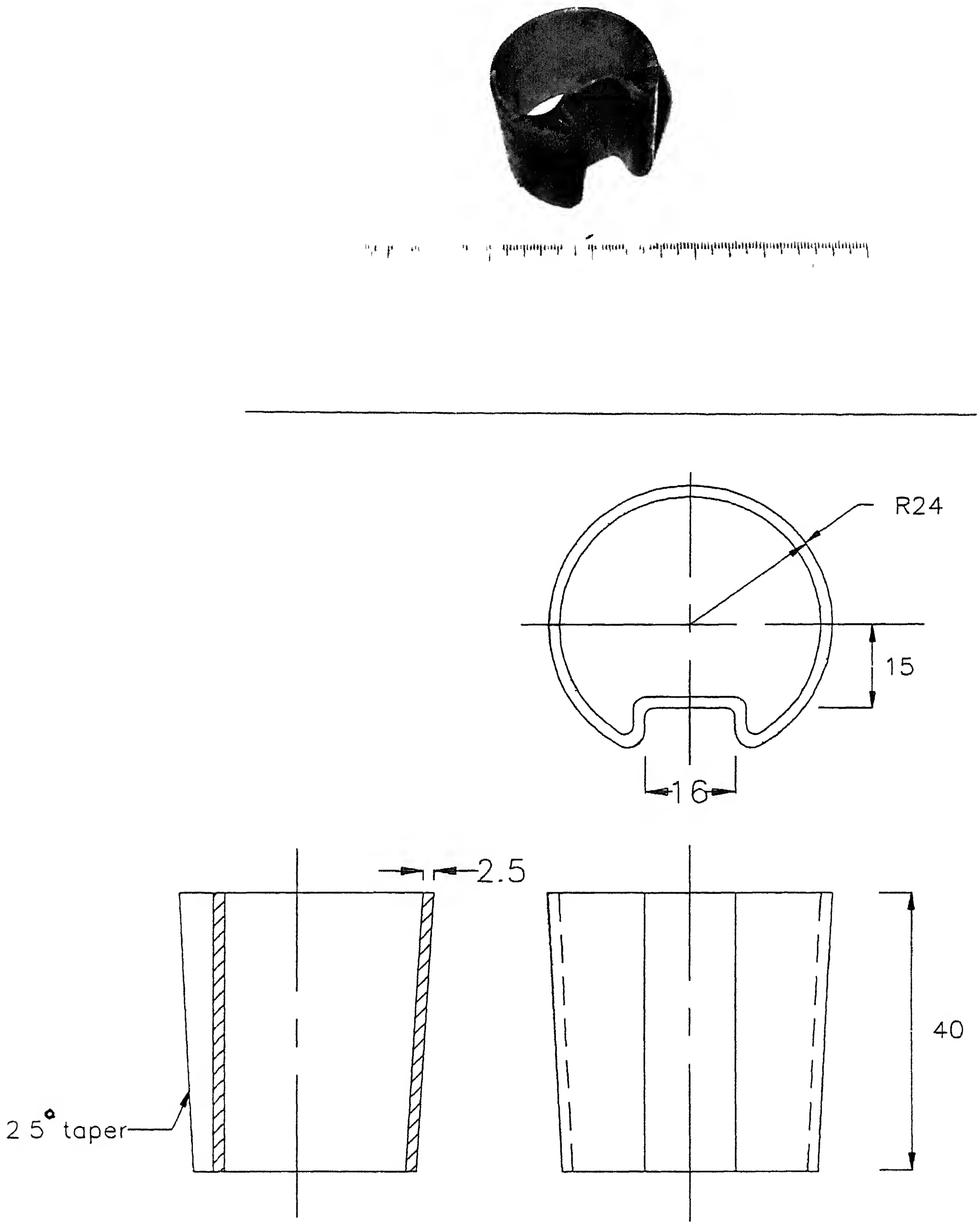


Figure 2.3 Photograph and drawing of the circular tube with square groove.

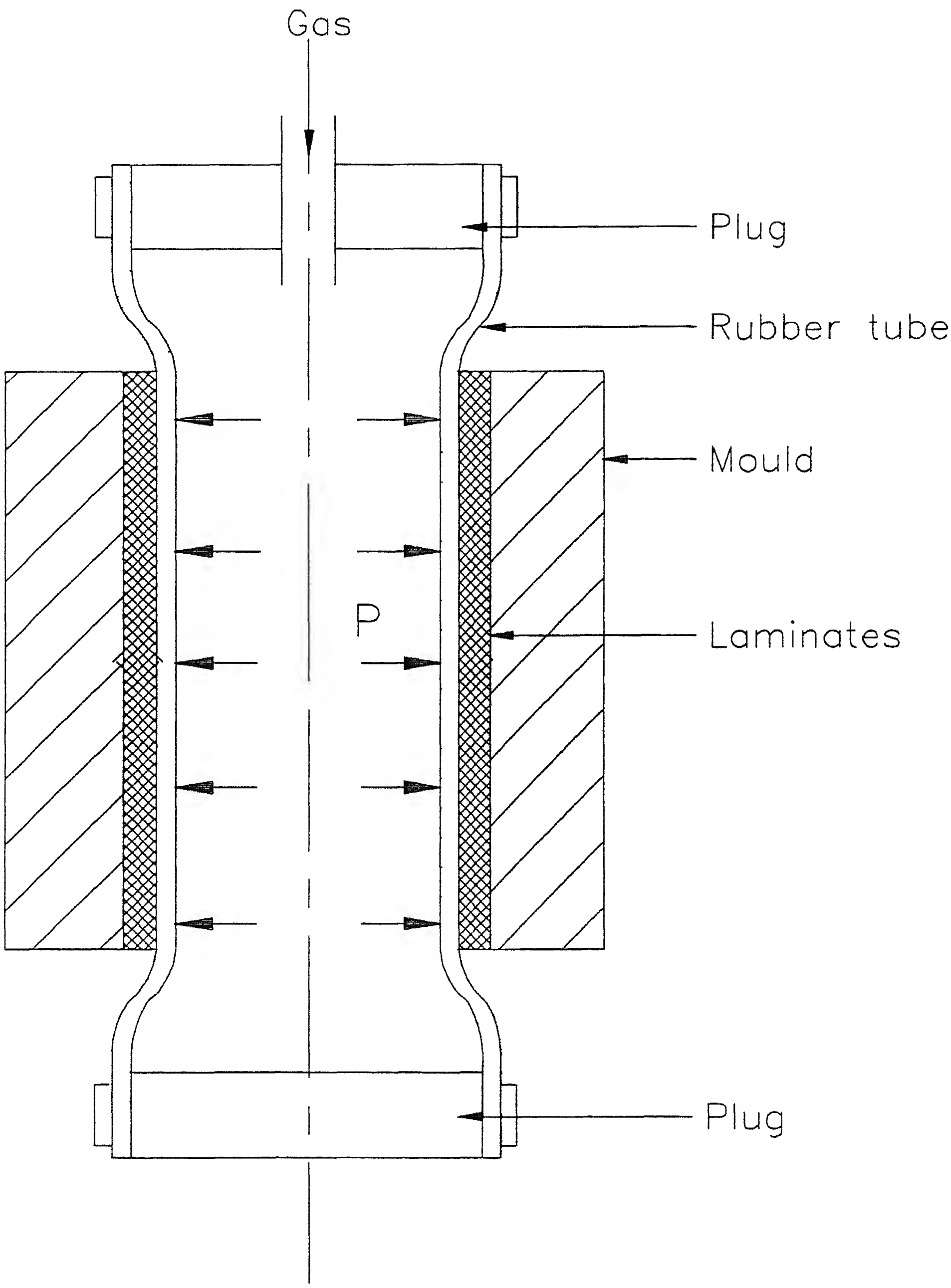


Figure 2.4 Principle of Pressure Moulding System

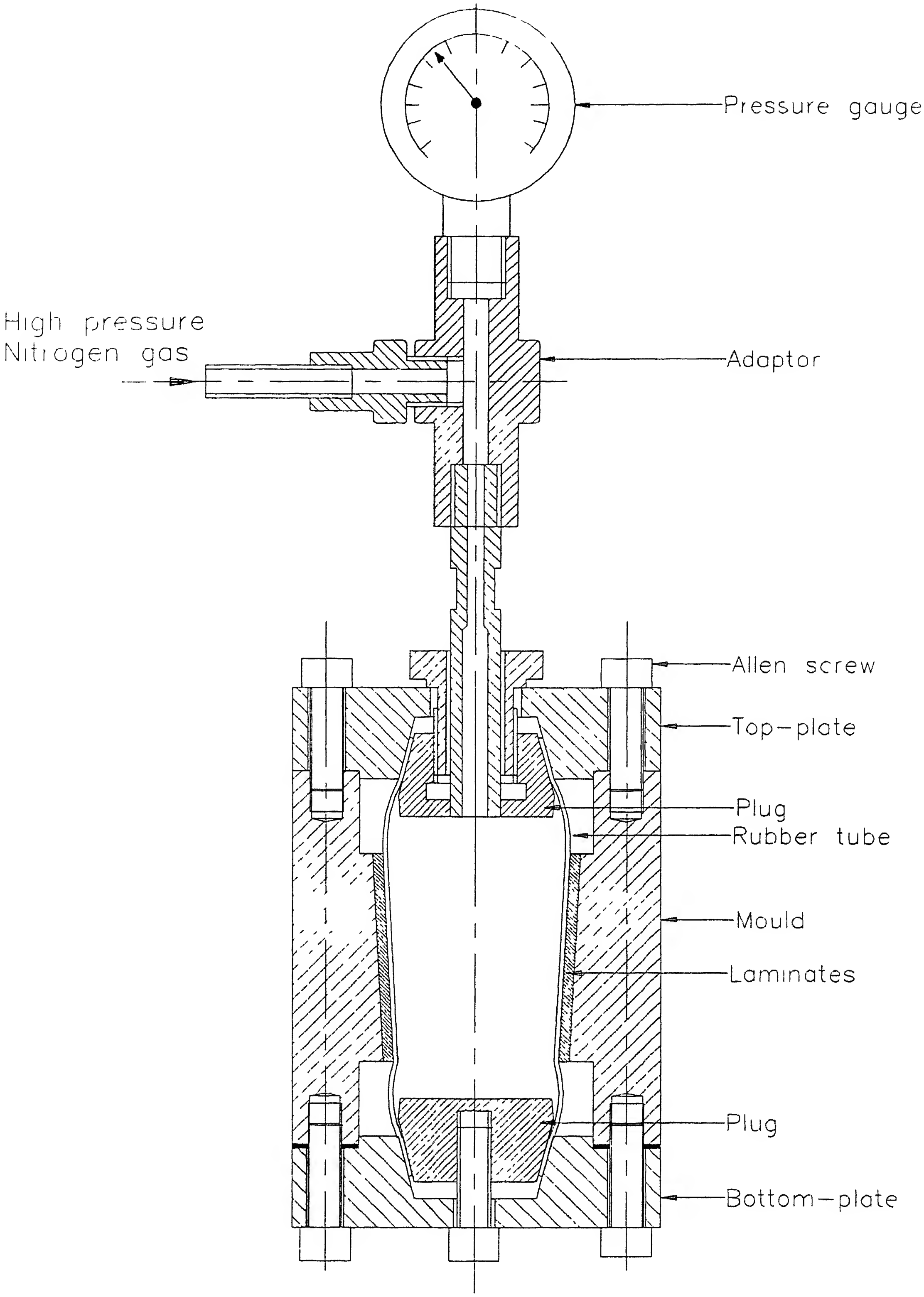


Figure 2.5 Half-sectional view of the Laboratory Scale Set-up

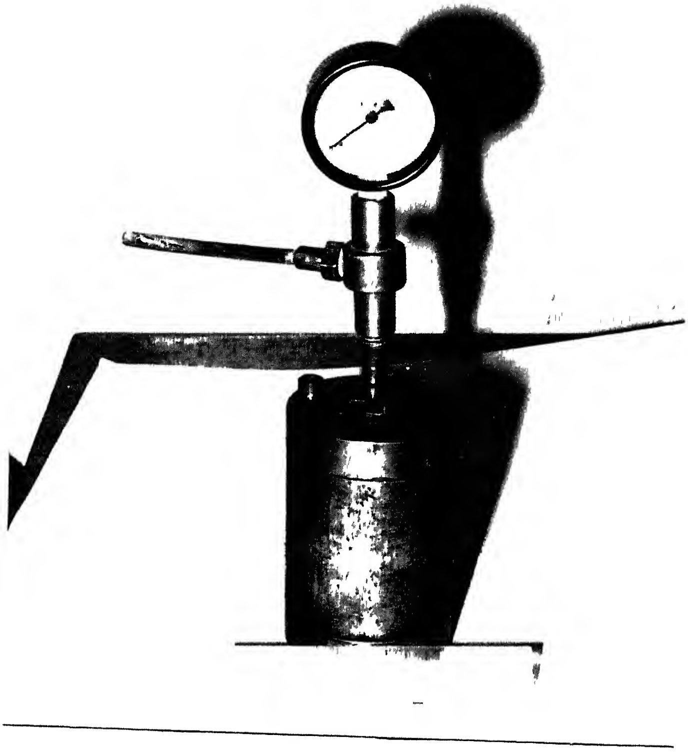


Figure 2.6 Photograph of the Laboratory Scale Set-Up.

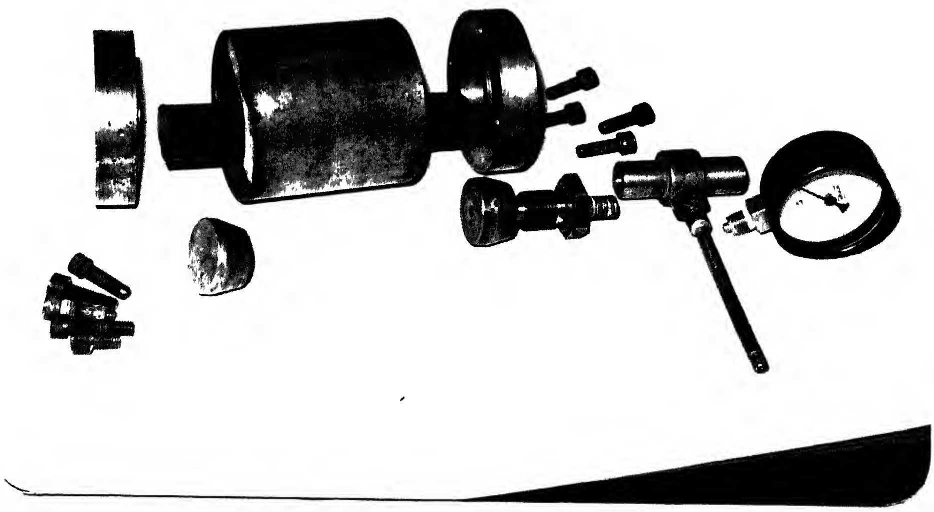


Figure 2.7 Photograph showing the exploded view of the Laboratory Scale Set-Up.

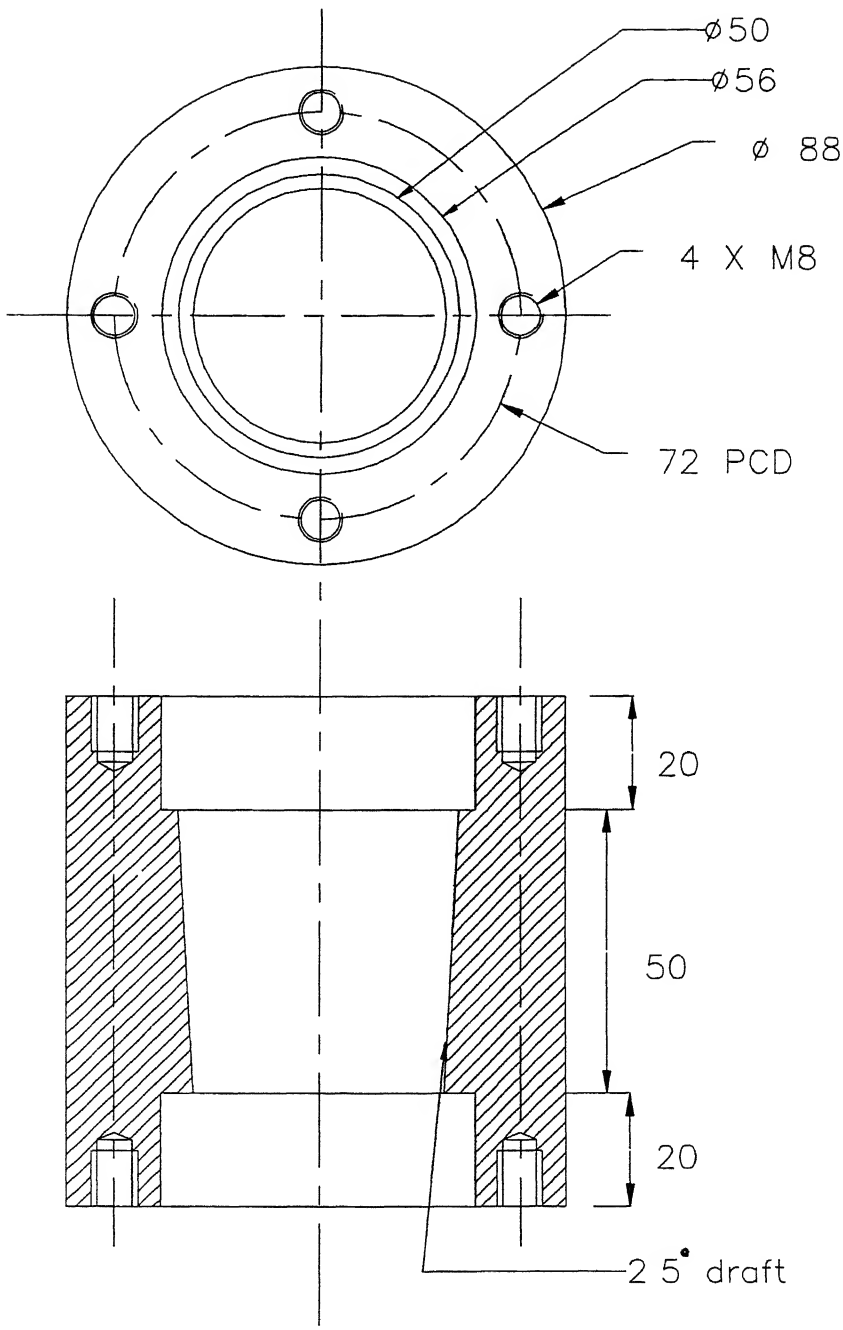
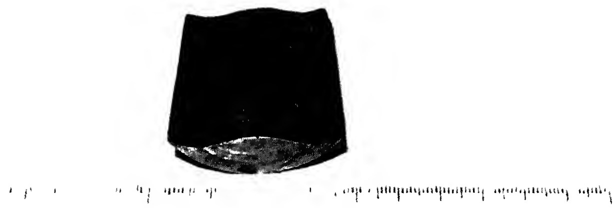


Figure 2 8 The Mould



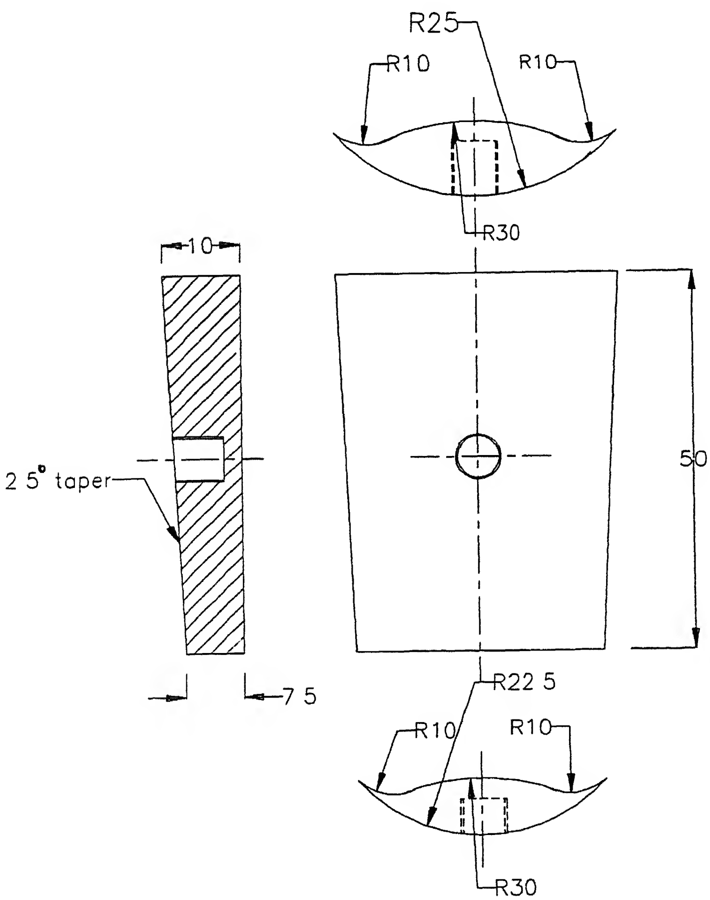


Figure 2.9 Photograph and drawing of The Insert for Convex–Concave surfaced product

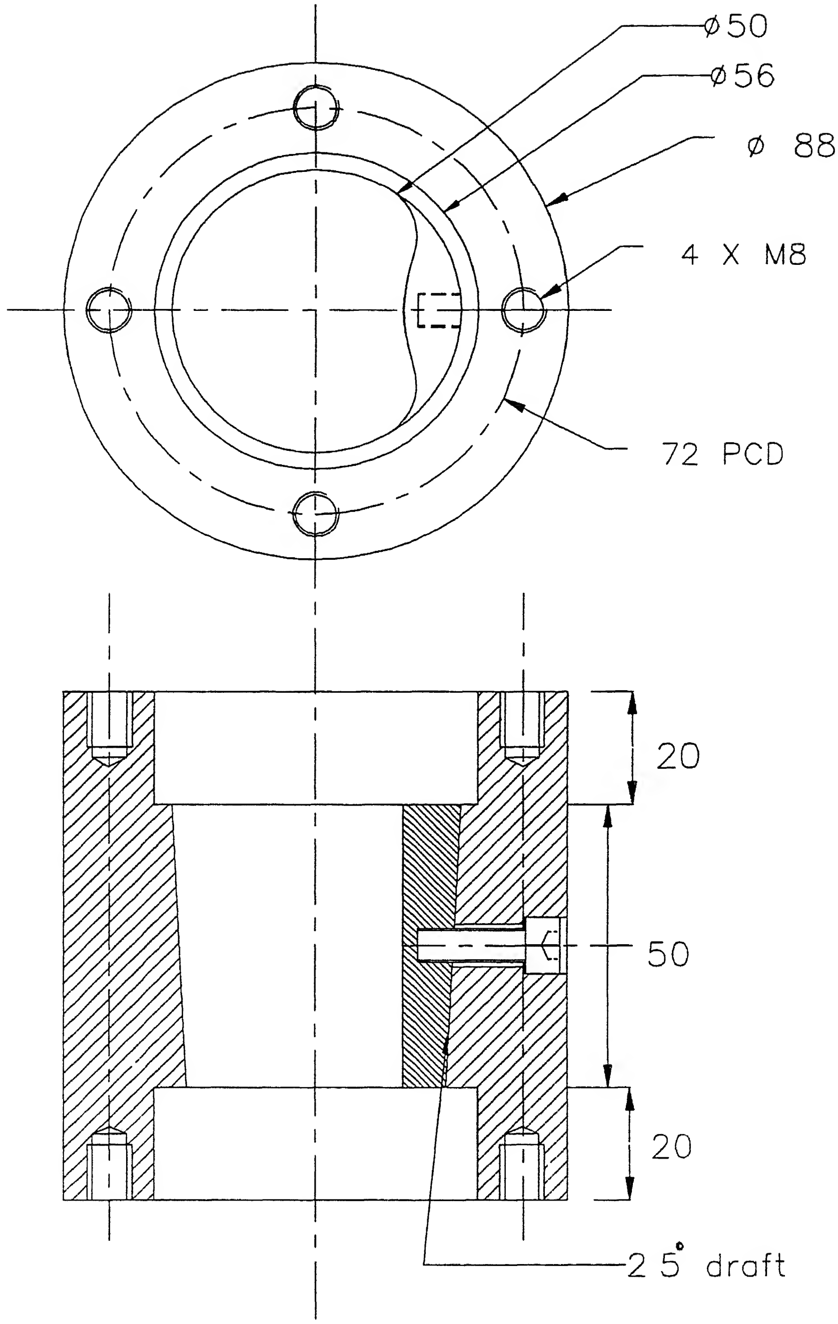


Figure 2 10 Mould for convex-concave surfaced product

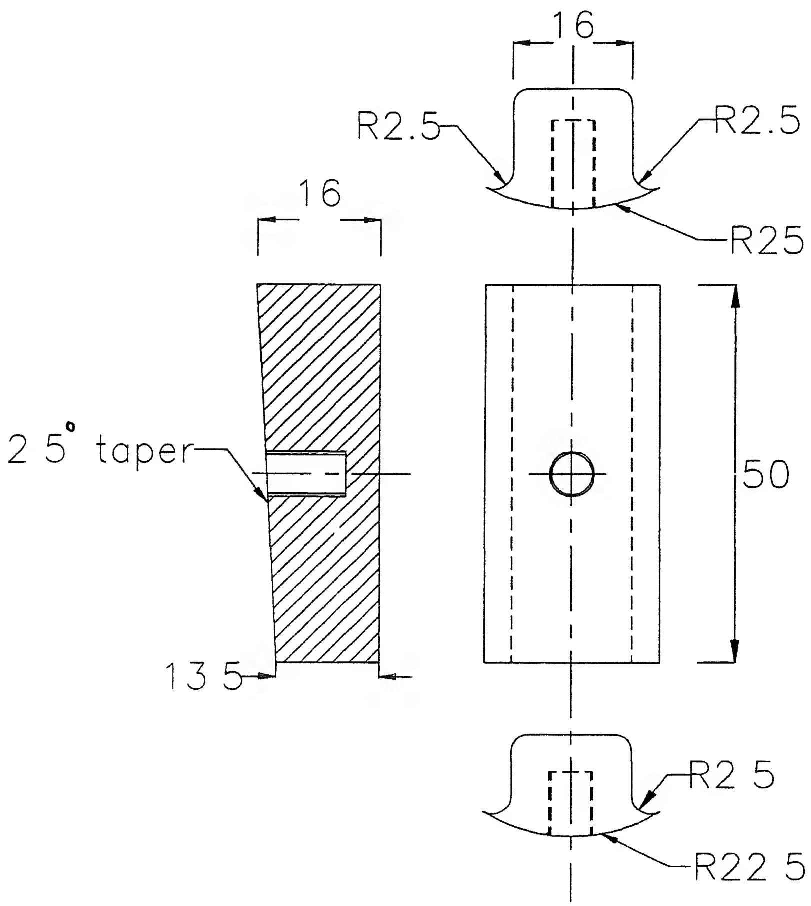


Figure 2 11 Photograph and drawing of the Insert for the Tube with a square groove

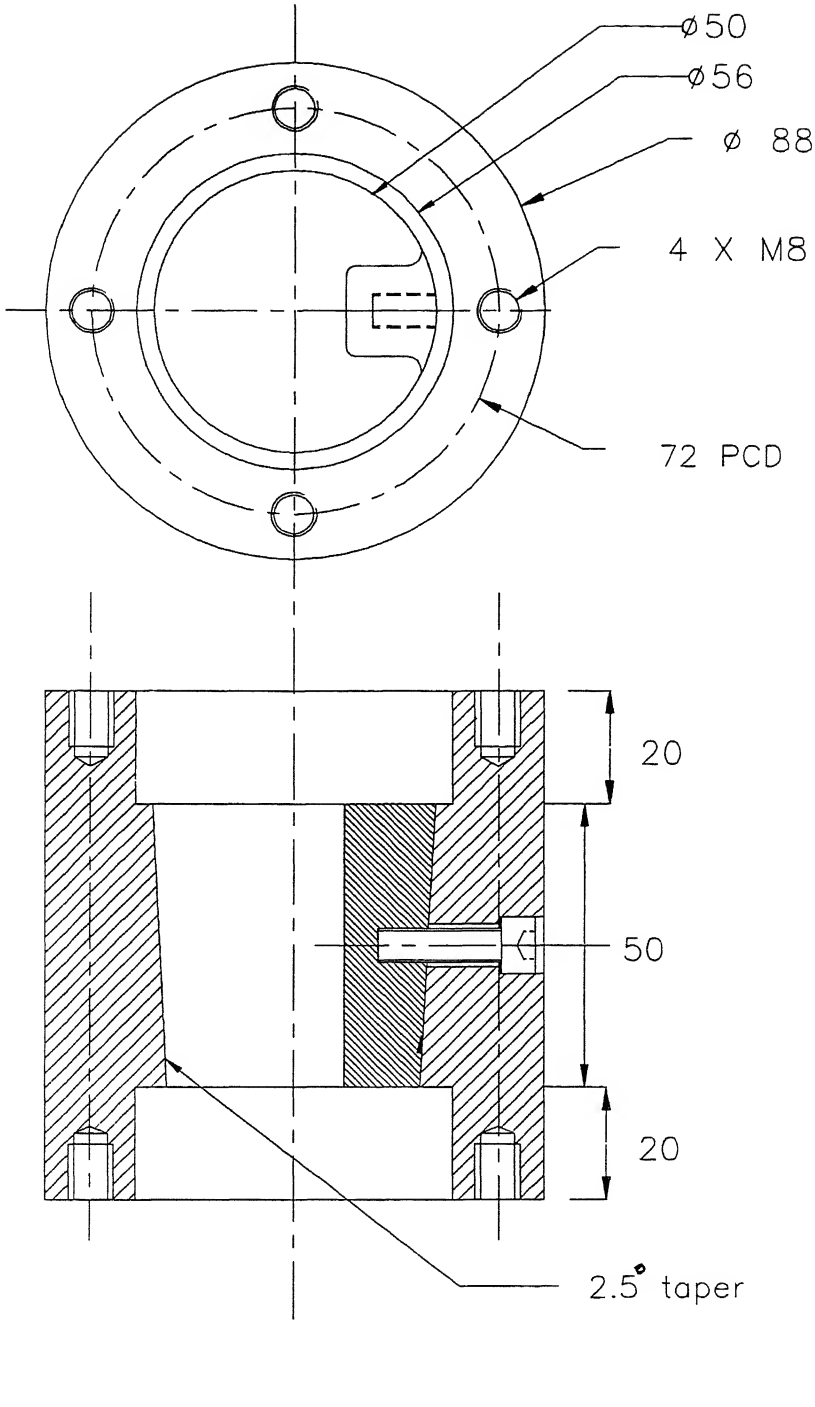
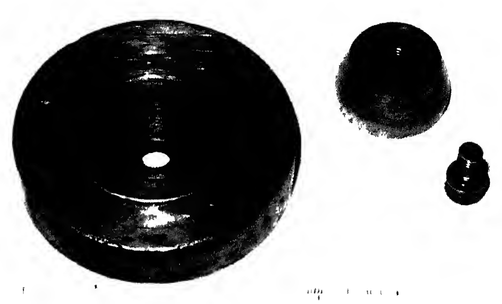


Figure 2.12 Mould for the tube
with a square groove.



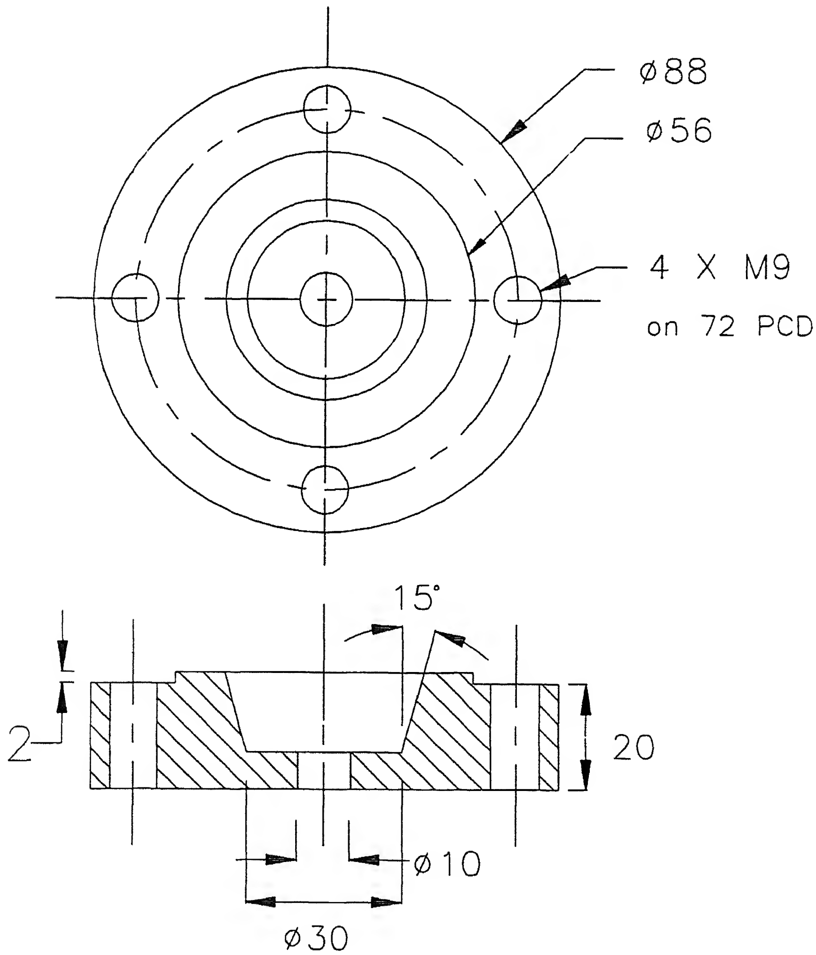


Figure 2.13 Photograph and drawing of The Bottom Plate

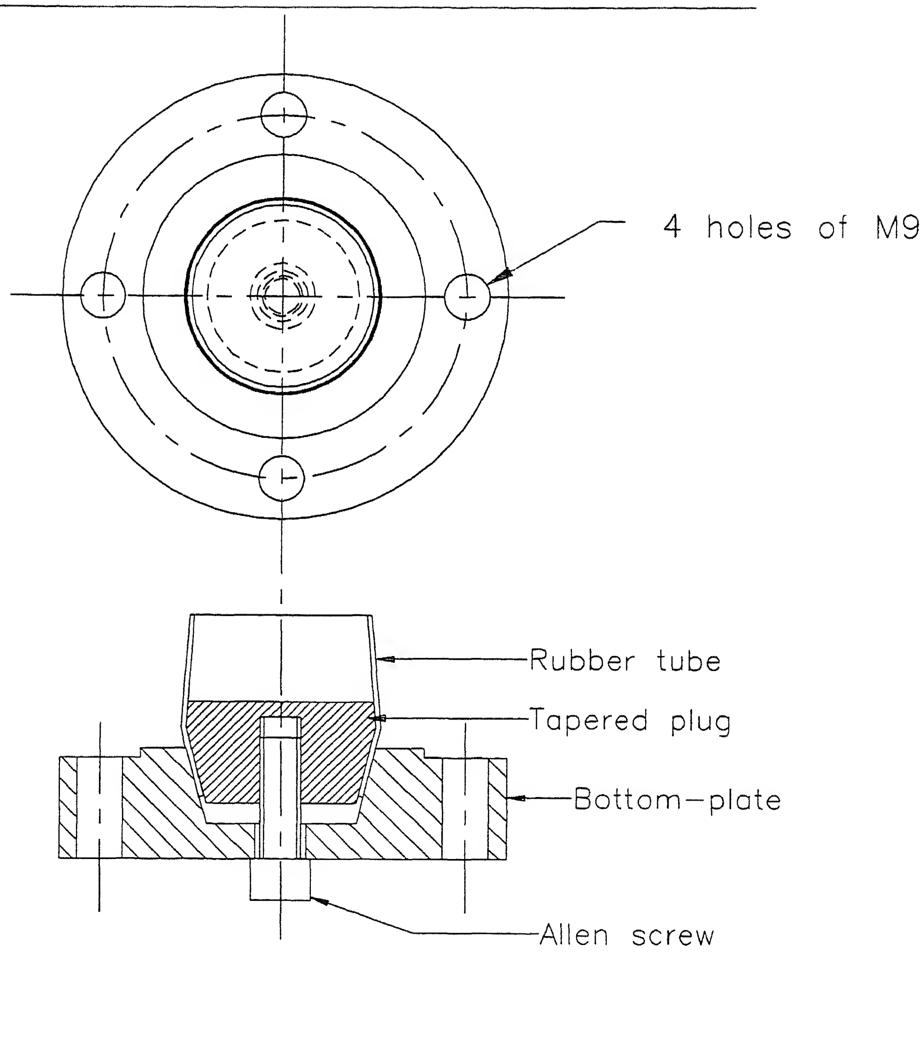


Figure 2.14 Photograph and drawing of the Bottom-Plate assembly

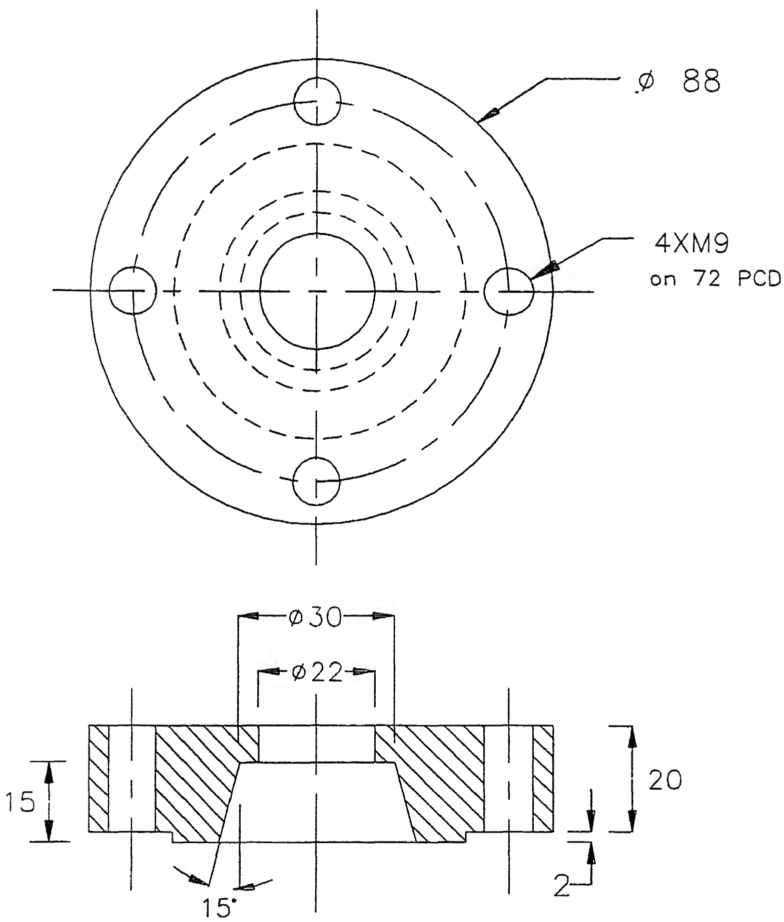


Figure 2 15 Photograph and drawing of the Top–Plate

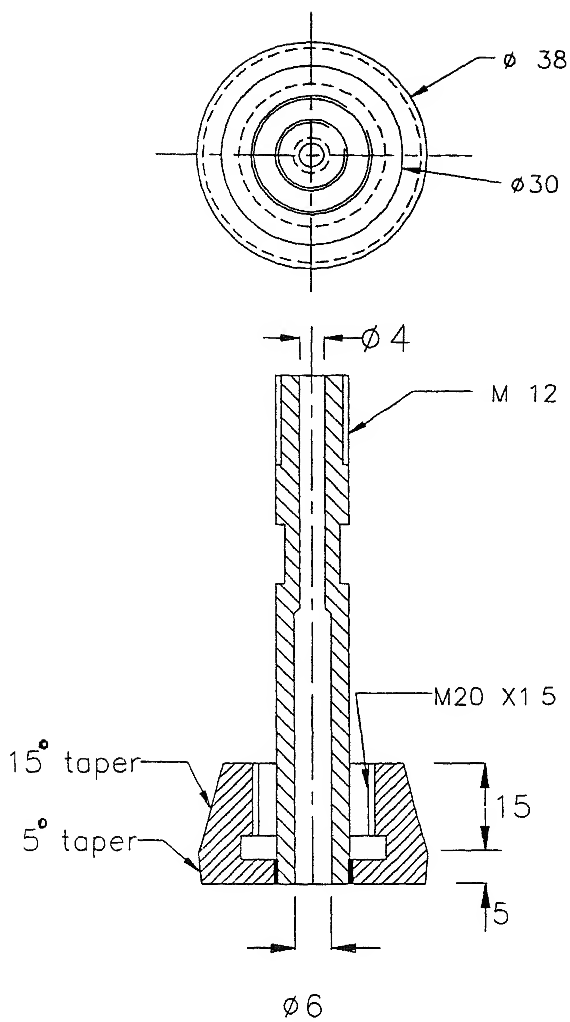


Figure 2 16 Tapered Plug of the Top-Plate

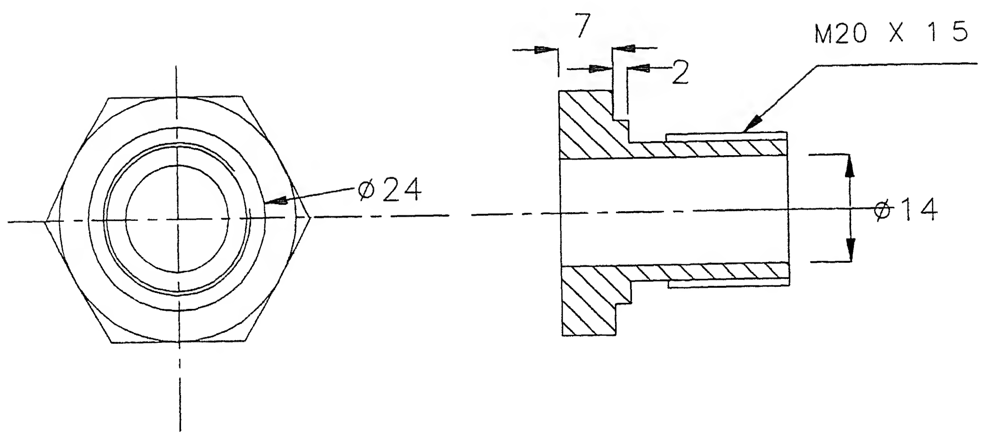


Figure 2 17 The Hollow screw

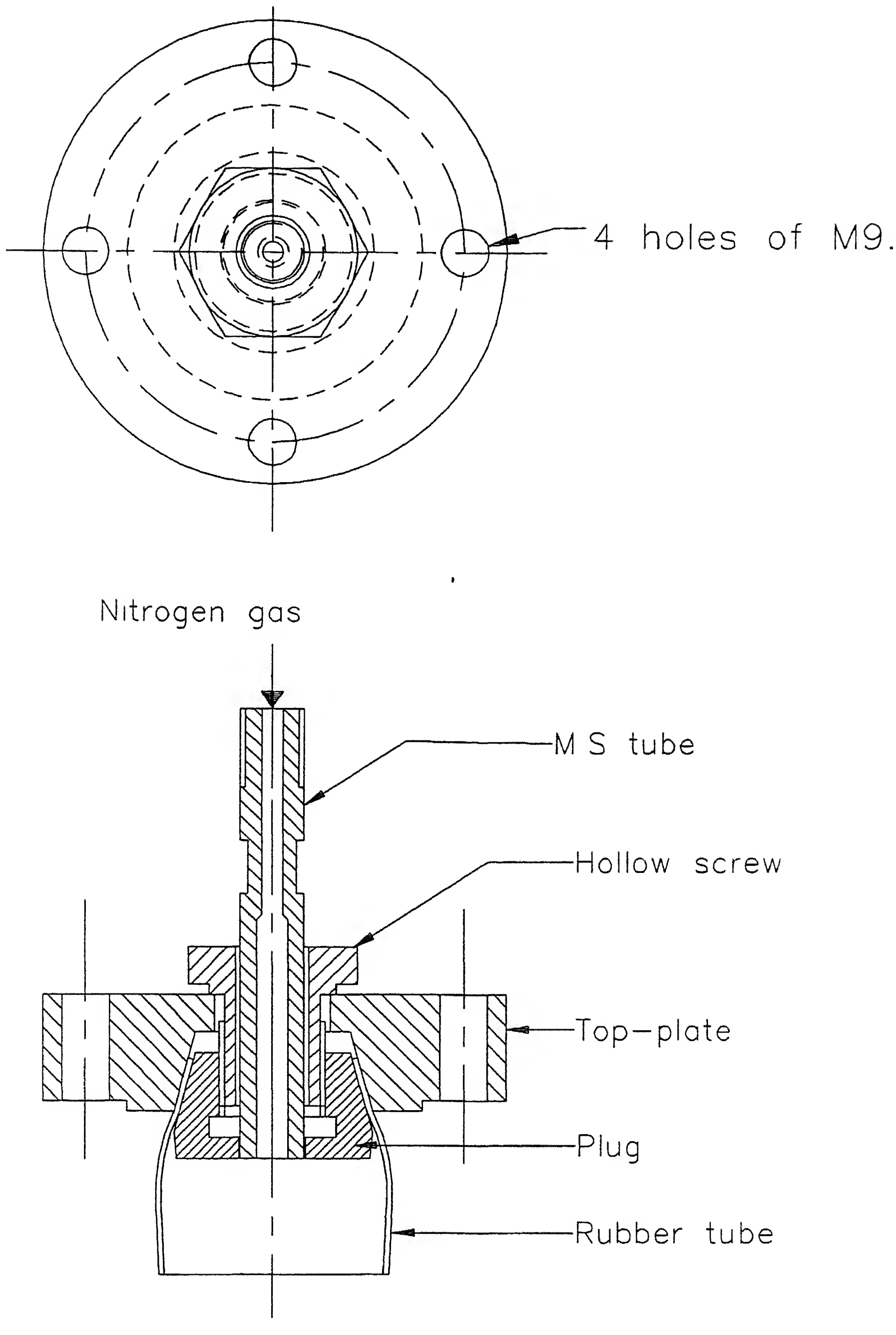


Figure 2 18 Top-Plate Clamping Mechanism

Figure 2.19 Photograph of the Top-Plate components.

Figure 2.20 Photograph showing the Top_Plate Assembly.

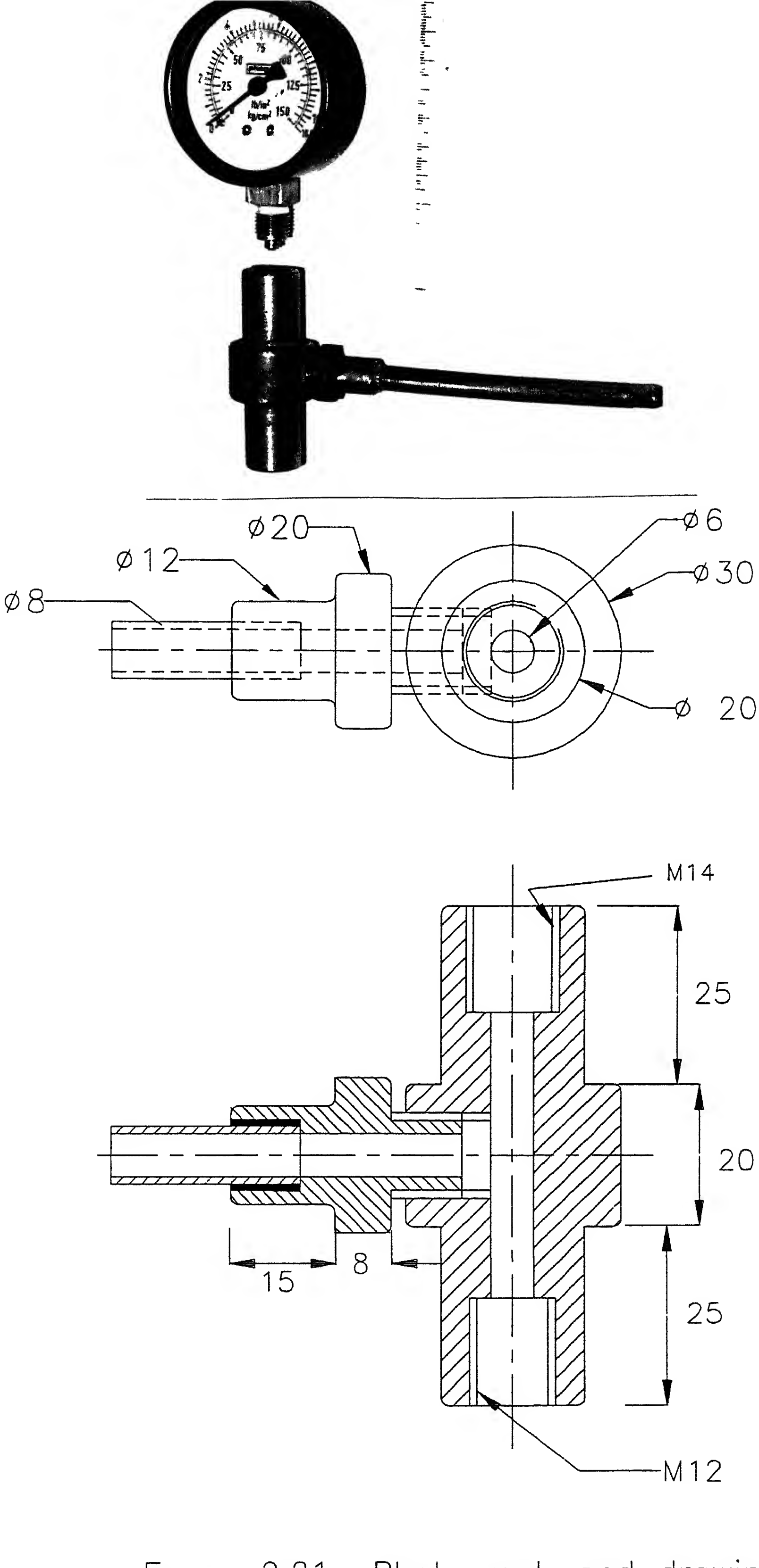


Figure 2 21 Photograph and drawing of the 3-Way Adaptor

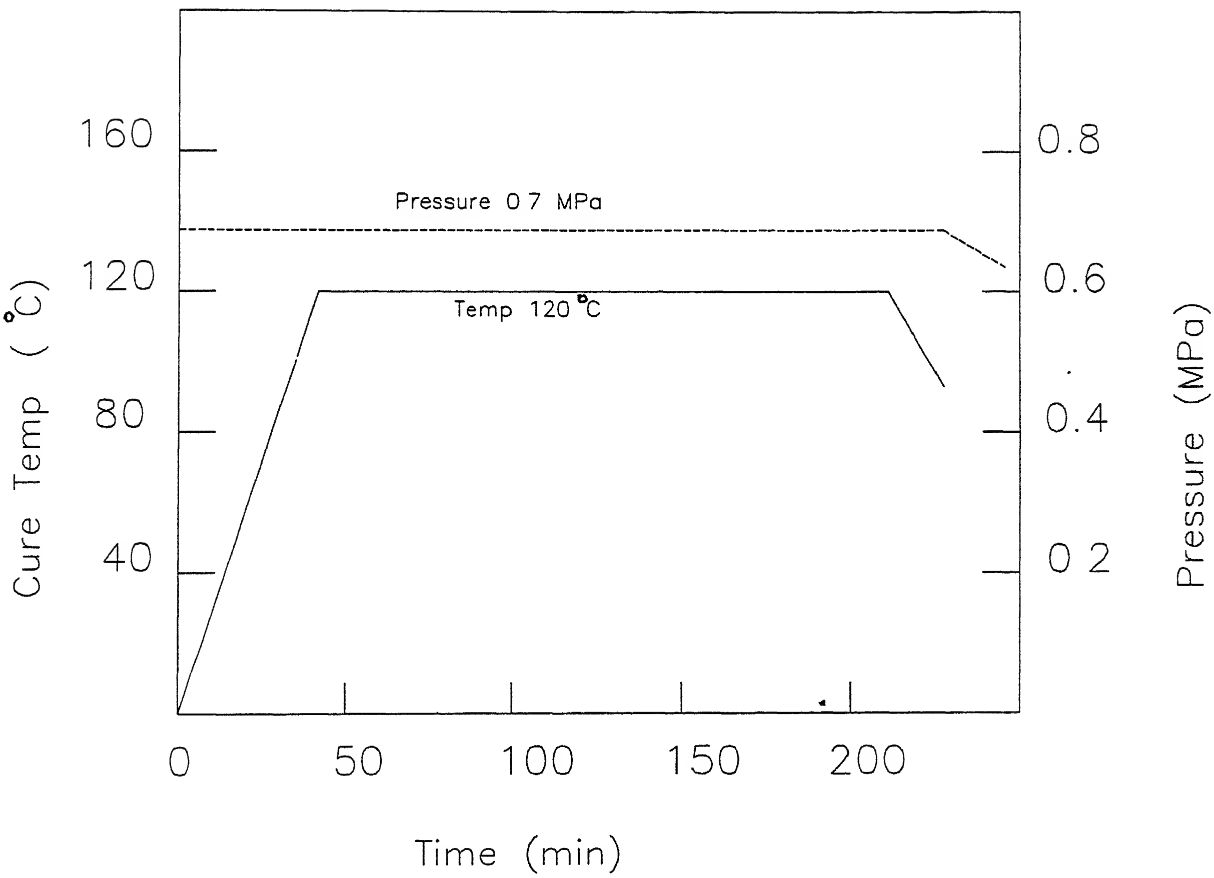


Figure 2.22 Illustration of the cure cycle used in the present work

CHAPTER 3

PRODUCT CHARACTERIZATION AND DISCUSSION

3.1 PRELUDE

The mechanical properties of a composite material are the most important data required for a designer to arrive at the physical dimensions of the end product. The mechanical properties of polymer composites can be estimated fairly closely, using the rule of mixture. But experimental proof is necessary to ascertain the validity of the theoretical determination of the properties.

Various fabrication parameters have the most significant influence on the properties of polymer composite materials. The curing cycle in particular imparts the mechanical properties in the composite. Tooling itself is another significant factor which affect the end mechanical properties. The curing pressure decides the fibre volume fraction. The curing temperature determines the duration of curing and imparts toughness properties.

3.2 OBJECTIVES OF CHARACTERIZATION

Characterization was carried out to evaluate the properties of the products fabricated through the Pressure Moulding System and hence the acceptability of the process. Several tests were conducted for the following:

1. Check the uniformity in thickness and circularity, and hence process capability.
2. Determine the fibre volume fraction of the products because many properties of

the final product are function of the volume fraction of the fibres and matrix.

3. Determine the mechanical properties such as ultimate flexural strength and modulus of elasticity.

3.3 PROPERTIES OF THE CONSTITUENT MATERIALS

The properties of the constituent materials greatly affects the nature and mechanical properties of the end product. Therefore they are discussed first before going to characterization aspect.

3.3.1 Glass Fibre

The fibre used in the present work is E-class glass fibre in woven form supplied by Ceat Ltd., Mumbai. Specifications of this fibre are given in Table 3.1.

Table 3.1 Specifications of Glass-fibre fabric

Fabric form	Untwisted, double weave
Nominal Thickness	0.44 mm
Area density	220 gm/m^2
Count	50-50, Bi-directional

3.3.2 Resin Material

The resin used is Epoxy resin Araldite LY 556 supplied by Hindustan Ciba-Geigy Ltd., Mumbai.

Araldite is a liquid unmodified diglycidyl ether of bi-shpenol 'A' (DGEBA) epoxy resin. Some of its physical properties are presented in Table 3.2 [Ciba 1994].

Table 3.2 Properties of Araldite LY 556

Epoxy content	5.2-5.45 equiv/Kg
Viscosity at 25°C	9000-12000 MPa s
Density at 25°C	1.1-1.2 gm/cm^3
Flash point	>200°C
Storage life	5 years.

The basic characteristics of epoxy resins are: easy cure, low shrinkage and high adhesive strength.

3.3.3 Formulation of Epoxy Resin Mixture

Epoxy resins are cured by means of a curing agent, often referred to as hardener. Hardener is added to basic resin to promote or control curing action taking part in it.

Hardener HY 951 (procured from Hindustan Ciba-Geigy Ltd., Mumbai) is used in this work. The properties of HY 951 are given in Table 3.3 [Ciba 1994].

Table 3.3 Properties of Hardener HY 951

Viscosity at 25°C	5000-8000 MPa s
Density at 25°C	1.1-1.2 gm/cm^3
Pot life after mixing with LY 556 at 25°C	0.75 - 1 hour

The impregnating solution prepared has the following composition.

Araldite LY 556 100 parts by weight

Hardener HY 951 10 parts by weight

This particular Epoxy-Hardener combination gives low cure shrinkage [Ciba 1992], and hence dimensional stability and small internal stresses in a product. Moreover, the products possess good mechanical and electrical properties. They are resistant to weathering and humidity. The products are not affected by most of the chemicals used in industries and have excellent water resistance. They do not cause corrosion and have low thermal conductivity and a low burning rate. They have good machinability

3.4 CHARACTERIZATIONOF CIRCULAR TUBE

3.4.1 Dimensional Stability

The diameter at both ends and thickness were measured using a vernier calliper. For measuring circularity, the tube was placed on a V-block. A dial indicator was set to zero, then the tube was slowly rotated and readings noted down. Table 3.4 provides the details of the dimensions achieved.

Table 3.4 Dimensions of circular tube

Specimen No	Avge. Outside Diameter (mm).		Avge. Thickness (mm)	Circularity (mm)	Thickness variation (mm)
	Big end	Small end			
T1	48	44.6	3.1	0.6	0.4
T2	47.9	45	3.5	0.8	0.55
T3	47.84	45	3.3	1.1	0.28
T4	48	44.8	3.2	0.6	0.35

The results showed that variation of circularity is within 1.1 mm, which is quite reasonable. Variation in thickness is quite large (0.55 mm). This variation is due to non-uniform pressure over the laminates. One of the probable reason for the non-uniform pressure is that the rubber tube used in this work was cut from an ordinary cycle

tube and it has non-uniform stiffness. Furthermore the hand lay-up was crude, with overlap of fibre mat at certain places.

3.4.2 Determination of Fibre Volume Fraction

The relative fraction of the matrix and reinforcing material is one of the important factors which determines the properties of composite material. It can be represented as the weight fractions or the volume fractions as follows [Agarwal 1990]:

Weight fraction of fibres(W) and matrix (W) is given by,

$$W_f = \frac{w_f}{w_c} \tag{3.1}$$

$$W_m = \frac{w_m}{w_c} \tag{3.2}$$

where, w_f = weight of fibre

w_m = weight of matrix, and

w_c = weight of composite material.

To find volume fractions from weight fractions, the density ρ_c, of the composite material must be obtained. The conversion between the weight fraction and volume fraction can be obtained by the following expression:

$$V_f = \frac{\rho_c}{\rho_f} W_f \tag{3.3}$$

$$V_m = \frac{\rho_c}{\rho_m} W_m \tag{3.4}$$

where, V_f and V_m are fibre and matrix volume fractions while ρ_c, ρ_f and ρ_m are densities of composite, fibre and matrix material respectively.

In present work, burn test is carried out to determine fibre volume fraction. In burn test, the specimen of laminate (approximately of area 10mm X 10mm) is weighed in air (W_{ca}) on a weighing machine which has an accuracy of 0.0001 gm. The weight of the same specimen in water (W_{cw}) is obtained. Then, the density of composite can be found as follows:

$$\rho_c = \frac{W_{ca}}{(W_{ca} - W_{cw})} \tag{3.5}$$

The specimen is placed in a furnace at a temperature of about 750°C to 800°C for 3 hours till the matrix is burnt out. The residue is cooled and reweighed to obtain weight of fibres, w_f. The fibre weight fraction and hence volume fraction can be determined as explained earlier.

The curing temperature and pressure determine the volume fraction, by controlling the resin bleed out. The fibre volume fraction of some of the specimens processed at different curing conditions are given in Table 3.5.

Table 3.5 Fibre Volume Fraction.

Sr.No	Specimen No.	Curing Temperature (°C)	Curing Pressure (MPa)	Fibre volume fraction (%)
1	T3	70	0.4	42.28
2	T4	70	0.4	41.75
3	T5	90	0.5	45.32
4	T6	120	0.7	48.65

3.4.3 Determination of Flexural Strength and Modulus

A circular ring of 15 mm width (Fig.3.1) was cut from the circular tube. The ring was loaded in diametral compression in an Instron testing machine in displacement control mode. The load and corresponding displacement were recorded.

For thin isotropic circular ring, under diametral compression, close form solutions are available using linear elastic approach [Roark 1966]. The loading diagram is shown in Fig. 3.2. Bending moment (M), for ring having its thickness much smaller than diameter is given by,

$$M = P R \, (0.3183 - 0.5 \, Z) \tag{3.6}$$

Maximum bending moment is

$$M_{max} = 0.3183\ P_{maxR} \tag{3.7}$$

where,

P = Load supported by ring and

R = Mean radius of ring.

The flexure strength (σ) is obtained as

$$\sigma = \frac{M \frac{h}{2}}{I} \tag{3.8}$$

where

h = Ring thickness and

I = Moment of inertia of cross-section.

The deflection (δ) is given by,

$$\delta = 0.149\ \frac{P\ R^3}{E\ I} \tag{3.9}$$

where

δ = Deflection and

E = Youngs modulus of elasticity.

For the measured collapse load P_{max}, flexural strength σ_{ult} can be determined through Eqs. 3.6 and 3.7. Modulus E is determined using Eqs. 3.8 from the linear portion of load-deflection curve.

The ring is symmetric, made up from a 0/90, 50-50, bi-directional woven glass cloth. So the material is orthotropic i.e., has identical properties in hoop (θ) and axial (Z) directions. Thus the circular ring is quasi-isotropic and hence the formulas for the flexural strength and modulus of elasticity for isotropic material are used to establish the flexural strength and modulus of the composite ring.

The load-displacement curve, shown in Fig. 3.3, provides the ultimate load supported by the circular ring. Table 3.6 shows the flexural strength and modulus obtained for three specimen rings.

Table 3.6 Flexural strength and modulus of circular ring

SR. No.	Specimen No.	Fibre volume fraction (%)	P_{ult} (N)	δ_{max} (mm)	σ_{ult} (MPa)	E (GPa)
1	R1	42.26	590	3	125.25	7.8
2	R2	40.82	625	2.3	135.75	10.2
3	R3	42.23	500	3.3	138.63	9.6

The average flexural strength of the circular ring is 133.21 MPa and modulus of elasticity 9.2 GPa. To compare the strength and modulus values, tensile test was conducted and will be discussed in next section.

3.4.4 Tensile Strength and Modulus of the Circular tube

A tensile test was carried out to have more reasonable assessment of the strength and modulus of the circular tube. A longitudinal strip of 6 mm width was cut from the composite tube. The width of the specimen was kept small to avoid curvature on the two longitudinal faces. The tensile specimen is shown in Fig 3.4. A strain gauge was fixed at the centre of the specimen using Araldite.

The test has been conducted on Instron machine. The cross-head speed is maintained at 1 mm/min. Using a strain gauge indicator, the strain at different load positions is measured. The ultimate load applied on the specimen is noted from the load verses displacement curve and tensile strength has been determined. The load-displacement curve and stess-strain curve are shown in Fig 3.5. The slope of the stress- strain curve gives the tensile modulus of the specimen. Table 3.7 presents the values of tensile strength, tensile modulus for various specimens along with their fibre volume fractions.

Table 3.7 Tensile strength and modulus of the specimens

Sr. No.	Specimen No.	Fibre Volume Fraction (%)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)
1	L1	45.32	171.4	17.13
2	L2	42.98	148.2	15.75

The failure mode observed was interface shear failure.

3.5 CHARACTERIZATION OF CONVEX-CONCAVE SURFACED TUBE.

This was first of the complex shapes produced by pressure moulding system. The characterization was carried out to determine the quality of the lay-up, curing and the degree of consolidation.

3.5.1 Visual Observations

The visual observations showed the surface to be well cured, with good surface finish. The form is well taken except at the change of radius. These regions are slightly resin rich. The mould being a two piece mould (Fig. 2.10), the change in radius was not uniform.

3.5.2 Dimensional Stability

The dimensional stability analysis showed a thickness variation of about 1.1 mm at the places where curvature jumped. The thickness was on higher side in the resin rich regions. Again the reasons for this variation is the rubber tube. The ordinary cycle tube is much stiffer and is unable to take deep curvature. Also overlap of fabric mat at certain places is another reason for the thickness variation.

3.5.3 Determination of Fibre Volume Fraction

The average fibre volume fraction obtained was 44 %. The resin rich regions were not taken into consideration in determining the fibre volume fraction.

3.6 CHARACTERIZATIONOF TUBE WITH SQUARE GROOVE

3.6.1 Visual Observations

The surface of the tube was observed to be well cured. The outer corners came out well. The inner corners were observed to be resin rich. As expected, the rubber tube failed to stretch the glass fabric into deep corners. This was more with 0-90° fabric when compared with ± 45° fabric.

3.6.2 Fibre Volume Fraction

The average fibre volume fraction obtained was 45.6%. Again, resin rich regions were not taken into consideration in determining the fibre volume fraction.

3.7 DISCUSSION

The variation in the thickness was 0.55 mm and circularity was within 1.1 mm. Some of the reasons were described in section 3.4.1. To overcome these shortcomings, the alternative ways are,

1. Using a thin walled rubber tube
2. Use of prepregs
3. Use of better lay-up procedure.

The fibre volume fraction obtained was 48.65 % (at 120°C and 0.7 MPa), which is marginally lower than fibre volume fraction of a plain laminate. For a woven fabric composite, fibre volume fraction of 50~55 % is considered to be optimum. The reasons for obtaining lower fibre fraction in this work are low pressure and short pot life of resin mixture. Higher fibre volume fractions (50 %) can be achieved by using curing pressure in the range of 0.8-1 MPa. This curing pressure should be applied before the gelation starts (pot life is about 45-50 minutes), otherwise excess epoxy will not bleed out.

Average flexural strength obtained was 133.21 MPa and modulus of elasticity 9.2 GPa. These are for an average volume fraction of 41.77 %. The average tensile strength and modulus of the longitudinal specimen obtained are 159.8 MPa and 16.44 GPa at fibre volume fraction of 44.15 %. The flexure strength of circular ring is 83.3 % of the tensile strength. Generally flexure strength is on lower side as compared to tensile strength.

3.8 CLOSURE

A reasonably good dimensional accuracy is achieved in case of circular cross-sectioned tube with circularity of 1.1 mm and thickness variation of 0.55 mm. Thickness variation is high, but it can be controlled by using more uniform rubber tube. The fibre volume fraction obtained in this work is quite reasonable. The convex-concave surfaced tube is

dimensionally stable except at sharp curvature of corner points.

The attempt to produce a circular tube with square groove, was partially successful. The outer surface of the composite tube was well taken. The inner surface was good except at the corner portions, where it was resin rich. Better results can be expected if the rubber tube is custom made to have cross-section same as that of the product.

The Pressure Moulding System, developed in this work, has potential to overcome some of the short comings of hand lay-up and bagging methods. As compared with the bagging methods, the tooling for this system is simple and much less expensive. Costly autoclave is not required and expensive bagging material required in pressure bag technique are eliminated. The Pressure Moulding System produces components reasonably well if the change in curvature is not abrupt.

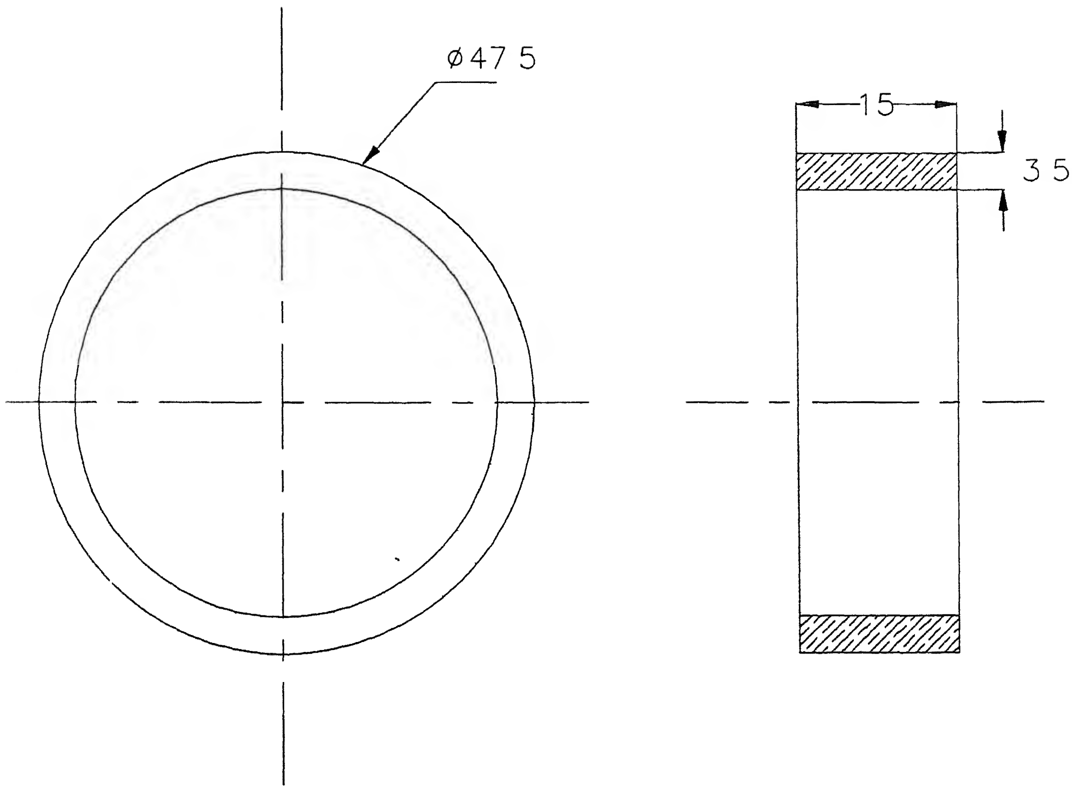


Figure 3 1 Geometry of Circular Ring Specimen

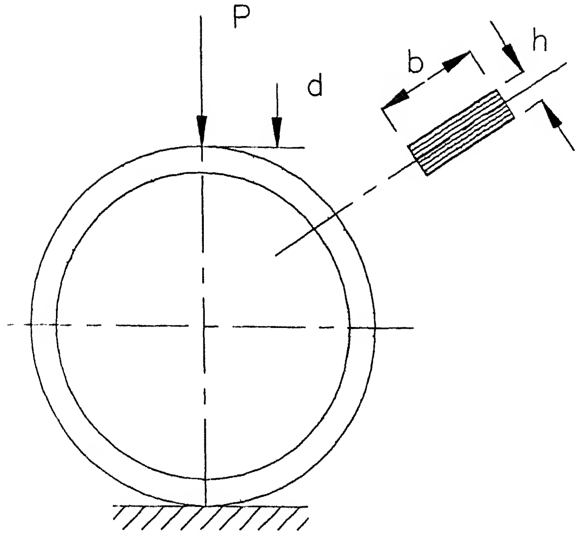


Figure 3.2 Schematic view of diametral loading

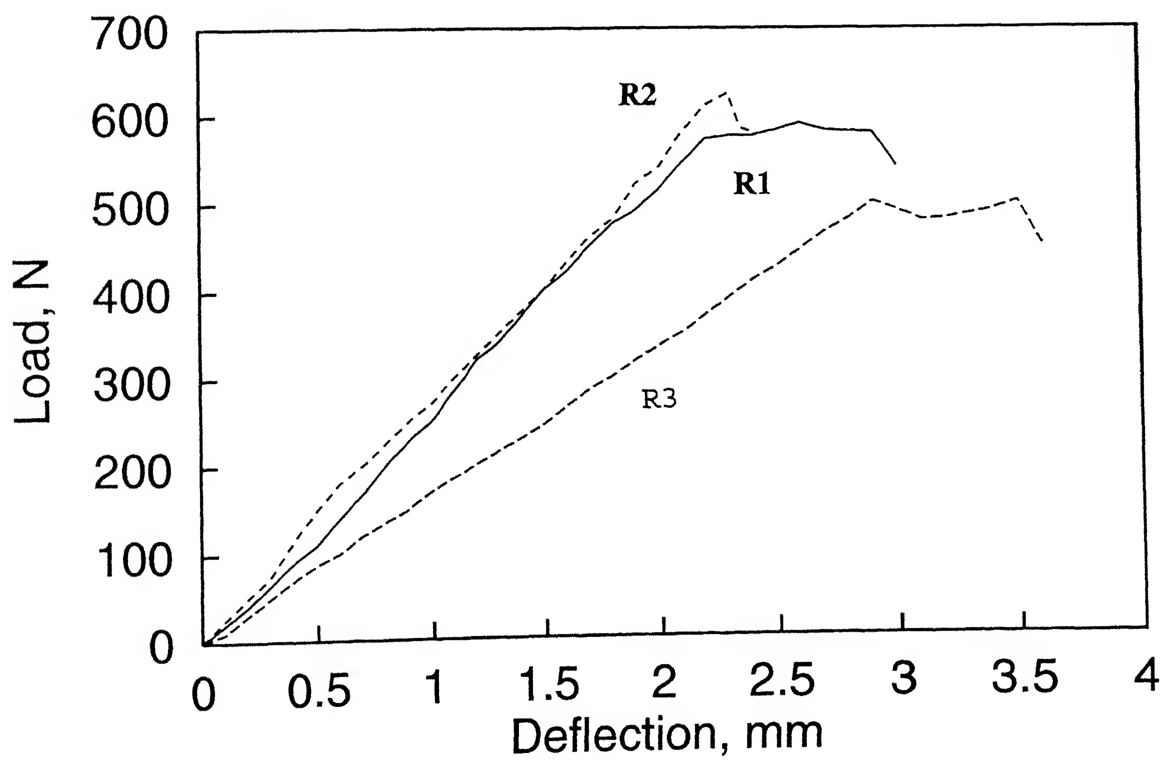
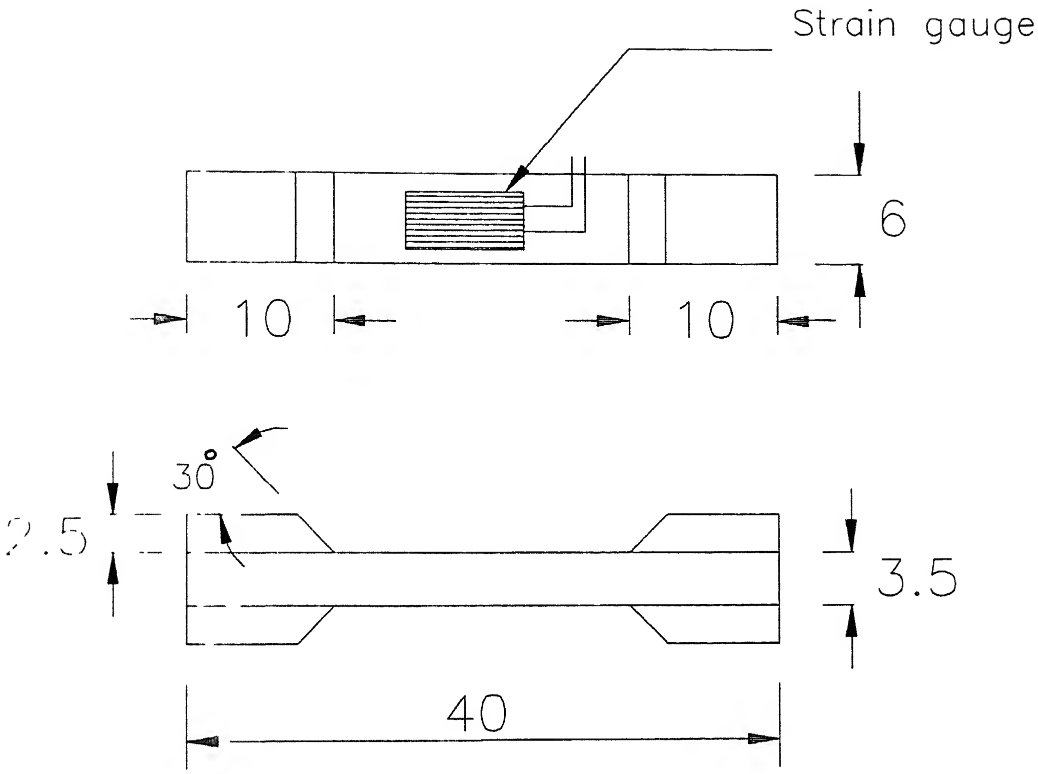


Figure 3.3 Load-Deflection Curve for the Ring specimen



All dimensions are in mm

Figure 3 4 Geometry of Tensile specimen

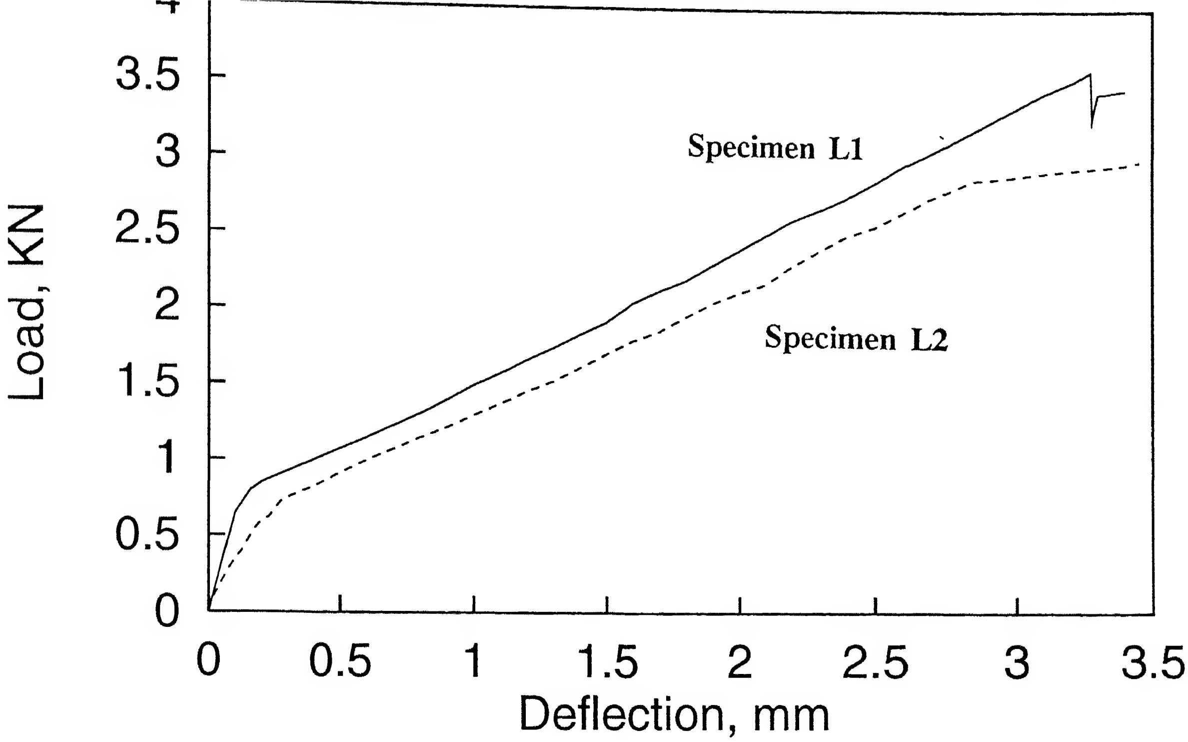


Figure 3.5 Load-Deflection curve of the tensile specimen

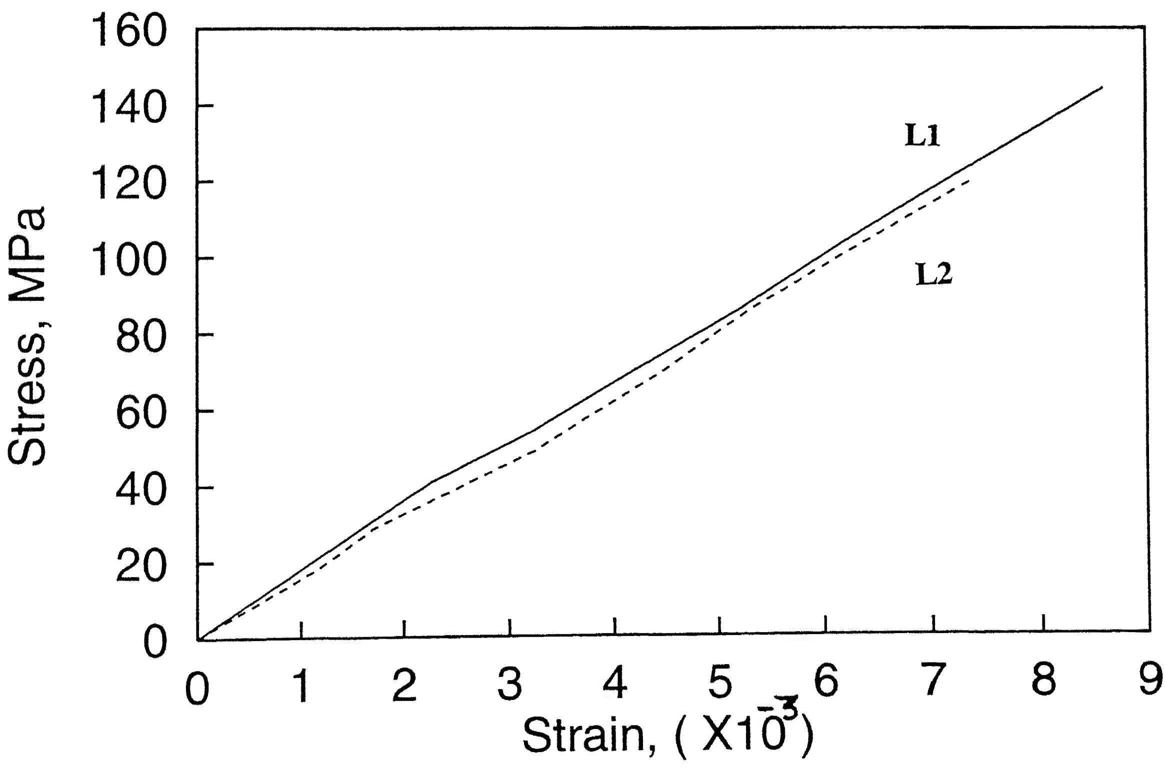


Figure 3.6 Stress-Strain Curve for the tensile specimen

CHAPTER 4

SUMMARY AND SCOPE FOR FUTURE WORK

4.1 SUMMARY OF THE PRESENT WORK

Development of an efficient tooling system plays a very important role in the realisation of composite products. To overcome some of the limitations of conventional techniques, a Pressure Moulding System was designed and fabricated to produce polymer composite materials. The main features of this system are:

* Simple yet effective design
* Less weight and easy handling
* Net moulding with least trimming of the product
* Easy assembly and removal of parts
* Low equipment cost, suitable even for small scale industries
* Low processing cost

Three different products fabricated and tested, have given satisfactory results. Experimental results are encouraging. Acceptable dimensional accuracy and stability was achieved. The products have reasonable strength, stiffness and fibre volume fraction.

The rubber tube, used in this work, turned to be the weak point. This tube cut from a bicycle tube is not appropriate because it is thick and stiff. Tube making technology

should be developed along with the pressure moulding system so as to make thin walled rubber tubes appropriate with the geometry of the product.

4.2 SCOPE FOR FUTURE WORK

The experimental results have boosted confidence and paved the way to venture into challenging tasks like

* Use of Prepreg tapes
* Moulding at higher temperatures and high pressure
* Fabricating products with complex geometries (e.g Gear profile).

REFERENCES

Agarwal B D, and Broutman L J, (1990), " Analysis and Performance of Fibre Composites ", John Willy and sons, New York.

Lubın Jorge, (1982), " Handbook of composites ", Van Nostrand, New York.

Hollaway Leonard,(1994), " Handbook of Polymer Composites for Engineers ", Woodhead Publishing Ltd., Cambridge, England.

Uday Kothalikar, (1996), " Balloon Compression Moulding- A New Technique for Fabricating FRP Products ", *M.Tech Thesis,* IIT Kanpur.

Loos A C, Springer G S, (1983), " Curing of Epoxy Matrix Composites ", Journal of Composite Materials, Vol.17, March 1983, 135-169.

Roark Raymond J, (1965), " Formulae for stress and strain ", Mcgraw-Hill Book Company, New York.

APPENDIX - A

GLASS TRANSITION TEMPERATURE OF THE RUBBER MATERIAL

The experimental set up to find glass transition temperature (T_g) of the rubber material used in the present work, is shown in Fig A-1. A rubber strip of 20 mm X 1.1 mm cross-section, one end fixed in water jacket, passes on two rollers. A 5 Kg weight was hung at the other end of the rubber strip. Water was filled in the jacket upto 90 mm length of the rubber strip, and heated gradually. For every 5°C raise in temperature, the elongation in the rubber strip was measured from the scale fixed behind the dead weight. Figure A-2 shows the 'temperature-thermal strain' curve of the rubber material, at an engineering stress of 2.23 MPa. From the curve, it was observed that the thermal strain is 26% at 70°C and it is 43% at 80°C. The specimen failed at about 90°C.

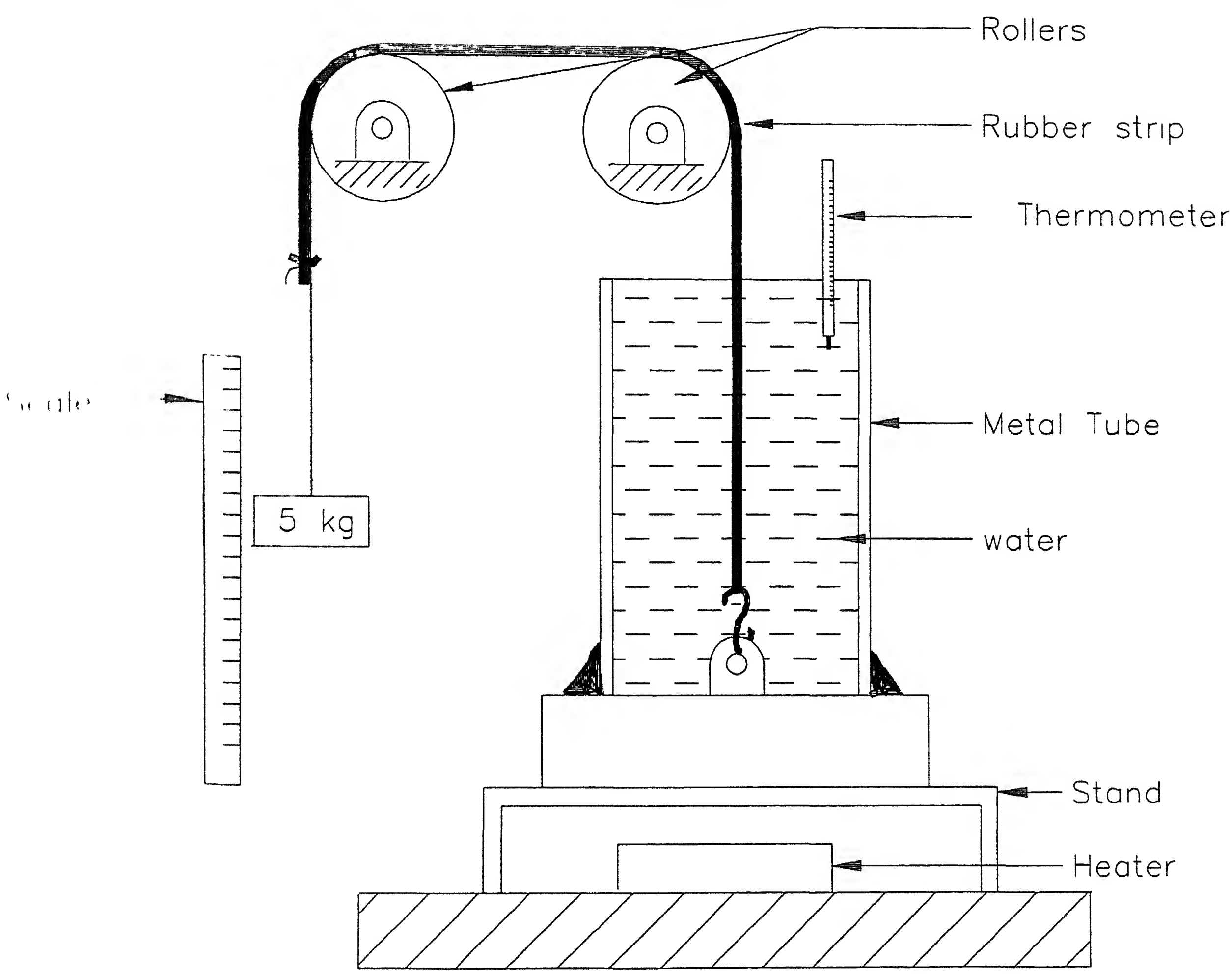


Figure A–1 Experimental set--up to find glass transition temperature of the rubber material

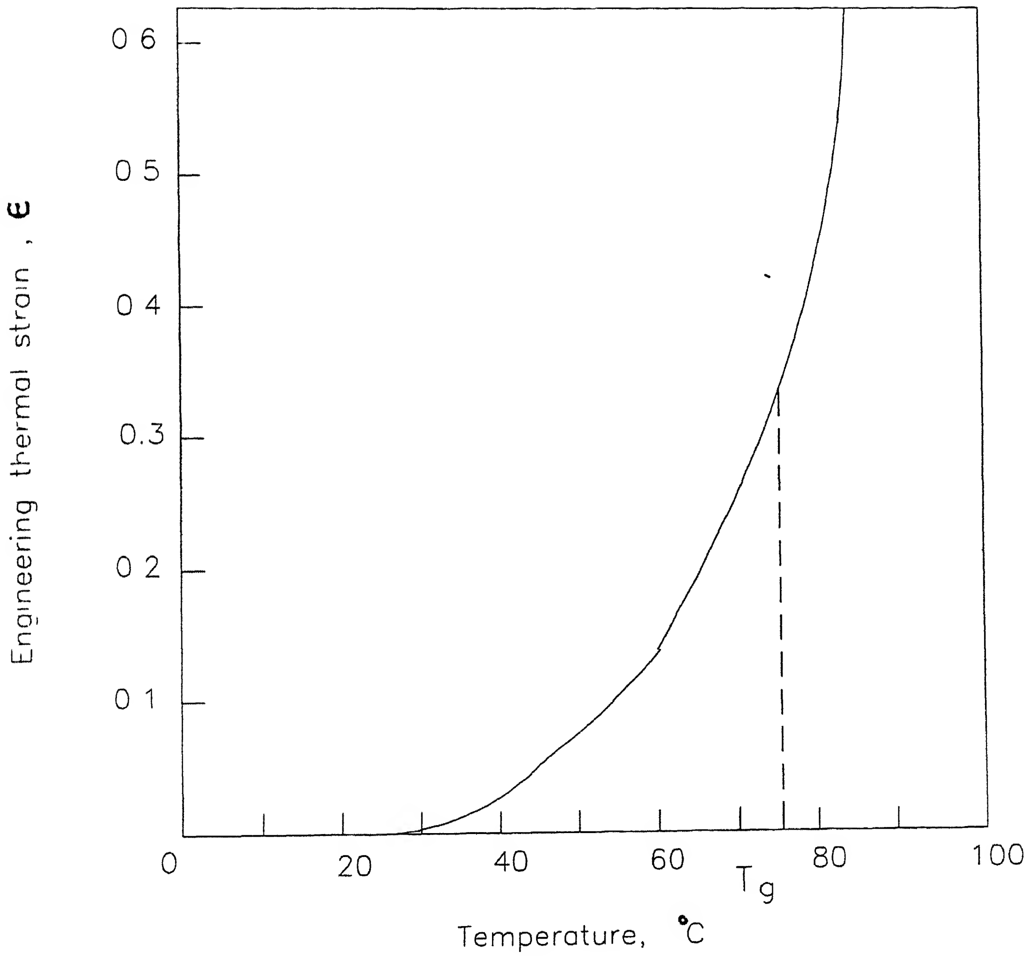


Figure A–2 Temperature – Strain curve for the rubber materi